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Vol. 112

MILLING METHODS

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Technical Committee on Milling Methods

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To

ROBERT HALLOWELL RICHARDS

Noted pioneer in mining education in America, prolific writer and inventor, indefatigable worker in research, leader in his profession, and a most gracious gentleman, beloved alike by his former students, by mining engineers, and by his fellow men, this volume is affectionately dedicated in recognition of his contributions to the art of ore dressing.

Preface

This volume is the outcome of a combination of fortunate circumstances, among which may be mentioned the generous action of the members of the Rocky Mountain Fund Committee in relieving the Institute of the problem of financing, the wealth of new material resulting from recent advances in the art, particularly along the line of flotation, the spontaneous response of authors to the announcement that a Milling Volume was to be published, and the liberal attitude of many companies in authorizing staff members to contribute papers and in releasing valuable data. Moreover, the preparation of Volume 106, on Copper Metallurgy, in 1933 by the Technical Committee on Reduction and Refining of Copper, set an ideal example for the Committee on Milling Methods.

Following the example of the volume on Copper Metallurgy, this volume aims to stress recent developments and present practices, touching upon past history only when particularly significant. It is hoped that the broad scope of the papers will appeal to a wide range of readers, from the man who is milling precious metals to the man who is marketing a low-grade nonmetallic product; from the student who is learning the rudiments to the experienced teacher and research man; and from the mill operator who does a most successful job in spite of the lack of a background of higher technical training to the scientist who revels in mathematical formulas and the latest chemical and physical theories.

When the papers had all been received, the Milling Committee found itself in the embarrassing situation of having more material available than could possibly be included in a single volume. To meet the problem it had to select from writings that were all good those that were believed to possess the broadest appeal. The judgment of the Committee on Papers and Publications was most helpful. It is hoped that means may be provided for the early publication of papers that it was found necessary to hold over from this volume.

It is impossible to mention the names of all those to whom the Milling Committee is under obligation for cooperation and assistance, but special thanks are due to the Institute staff for taking care of the vast amount of editorial work involved; to Arthur F. Taggart, who, as Vice Chairman, devoted to the task an immense amount of time, and whose advice was invaluable; and to R. M. del Giudice, who acted as an unpaid and unofficial secretary in keeping track of the voluminous correspondence and routine details, a labor of love which he performed most effectively.

Charles E. Locke,
Chairman of Committee
on Milling Methods.

Cambridge, Mass.
November 15, 1934

Contents

Page
Committee on Rocky Mountain Income 3
Committee on Milling Methods. 3
A.I.M.E. Officers and Directors. 4
Preface. By Charles E. Locke 5
Robert Hallowell Richards. By W. Spencer Hutchinson. 11

PAPERS

Crushing and Grinding

The Hadsel Mill. By R. G. Hall 15
Ball Milling. By A. M. Gow, M. Guggenheim, A. B. Campbell and W. H. Coghill (With Discussion). 24
Advantage of Ball (Rod) Mills of Larger Diameters and Advantage of Improving Bearings. By Will H. Coghill, Fred D. DeVaney and R. G. O'Meara (With Discussion) 79
Some Fine-grinding Fundamentals. By A. W. Fahrenwald. 88
Summary of Investigation on Work in Crushing. By John Gross 116
Grindability of Various Ores. By Walter L. Maxson, F. Cadena and Fred C. Bond 130
Crushing and Grinding Characteristics as Determined from Screen Analyses. By Fred C. Bond and Walter L. Maxson 146

Classification and Settling

Fine Grinding and Classification. By J. V. N. Dorr and Anthony Anable 161
Colloidal Chemistry of Pulp Thickening. By Elliott J. Roberts 178

Flotation Theory and Practice

Principles of Flotation, I—An Experimental Study of the Effect of Xanthates on Contact Angles at Mineral Surfaces. By I. W. Wark and A. B. Cox (With Discussion). 189
Principles of Flotation, II—An Experimental Study of the Influence of Cyanide, Alkalis and Copper Sulfate on the Effect of Potassium Ethyl Xanthate at Mineral Surfaces. By I. W. Wark and A. B. Cox 245
Principles of Flotation, III—An Experimental Study of the Influence of Cyanide, Alkalis and Copper Sulfate on the Effect of Sulfur-bearing Collectors at Mineral Surfaces. By I. W. Wark and A. B. Cox 267
Hypothesis for the Nonflotation of Sulfide Minerals of Near-colloidal Size. By A. M. Gaudin and Plato Malozemoff. 303
Reactions of Xanthates with Sulfide Minerals. By A. M. Gaudin, Franklin Dewey, Walter E. Duncan, R. A. Johnson and Oscar F. Tangel, Jr. 319
The Case for the Chemical Theory of Flotation. By A. F. Taggart, G. R. M. del Giudice and Othon A. Ziehl 348
Action of Alkali Xanthates on Galena. By T. Clinton Taylor and A. F. Knoll 382
Study of Slime-coatings in Flotation. By G. R. M. del Giudice (With Discussion) 398
Production of High-grade Concentrate from Butte Copper Ores—Results of Laboratory Investigations. By B. S. Morrow and G. G. Griswold, Jr. 413
Microscopy in Flotation Research. By G. R. M. del Giudice. 424

PAGE

Flotation of Nonsulfides

Soap Flotation of the Nonsulfides. By WILL H. COGHILL AND J. BRUCE CLEMMER (With Discussion). 449
Milling Methods and Costs at No. 2 Concentrator of the Phosphate Recovery Corporation, with an Appendix on New Developments in the Flotation Concentration of Phosphate Rock. By H. S. MARTIN 466
Relative Floatability of Silicate Minerals. By JOHN MARK PATEK (With Discussion) 486

Magnetic Concentration

Magnetic Concentration of Ores. By R. S. DEAN AND C. W. DAVIS 509

Gold and Silver Milling and Cyaniding

Economical Techniques in Treatment of Gold Ore. By A. W. ALLEN. 538
Ore Treatment as a Factor in Small Gold-mining Enterprises. By ERNEST GAYFORD. 549
Increasing Gold Recovery from Noranda's Milling Ore. By C. G. MCLACHLAN 570
Review of Black Hills Metallurgy, with Particular Reference to the Homestake Ores. By ALLAN J. CLARK. 597
Milling Methods of the Porcupine District of Northern Ontario. By P. D. P. HAMILTON 617
Cyanidation at Kirkland Lake. By DONALD F. IRVIN. 646
Dissolution of Gold and Silver in Cyanide Solutions. By GEORGE BARSKY, S. J. SWAINSON AND NORMAN HEDLEY 660
Milling Gold Ores at Pioneer. By RUSSELL SPRY AND PAUL SCHUTZ 678
Ore Treatment at Beattie Gold Mines Limited. By W. G. HUBLER AND F. J. MARTIN 690
Milling Methods and Costs at Presidio Mine of the American Metal Co. of Texas. By VAN DYNE HOWBERT AND FRED E. GRAY. 704
Milling and Cyanidation at Pachuca. By R. R. BRYAN AND M. H. KURYLA. . 722
Cyanide Milling Practice of The Fresnillo Company. By W. E. CRAWFORD 734
Milling Methods at the Concentrator of The Fresnillo Company. By W. A. BINSACCA 751
Development of Milling and Cyanidation on the Witwatersrand. By F. WARTENWEILER. 760

Milling Practice—Nonmetallic Minerals

Mechanical Preparation of Nonmetallic Minerals. By PAUL M. TYLER 785

Milling Practice—Iron, Tungsten and Base Metals

Iron-ore Beneficiation in the Lake Superior District. By E. W. DAVIS 824
Concentration of Tungsten Ore by the Nevada-Massachusetts Co. By OTT F. HEIZER. 833
Milling Practice at Buchans Mine, Buchans, Newfoundland. By G. A. HELLSTRAND AND P. W. GEORGE. 841
Milling Practice in the Tri-State Zinc-lead Mining District of Oklahoma-Kansas and Missouri. By S. J. BURRIS, JR., C. O. ANDERSON, ROBERT E. ILLIDGE, WARREN HOWES AND M. D. HARBAUGH 861
Nkana Mine Concentrator. By L. P. DURHAM. 925
Concentrating Operations at the Roan Antelope Copper Mines Limited. By J. W. LITTLEFORD 935
Concentration of Polish Bleischarley Ores. By L. P. DAVIDSON AND M. C. MESSNER. 952
INDEX 965

Robert Hallowell Richards

The purpose to honor Professor Richards brings honor to all who participate in the preparation of this book. Whether for his matchless contribution to the teaching and training of youth in the fields of mining engineering and metallurgy; or for his splendid achievements in the specialty he has made his own—the mechanical concentration of ores; or for his loyal services to the American Institute of Mining and Metallurgical Engineers; or for his fine qualities of character, rugged simplicity, generosity and kindness, we hail him a gentleman without a peer, loved and admired by all who know him.

Professor Richards was born August 26, 1844, at Gardiner, Maine. From thirteen to eighteen years of age, young Richards attended school in England, the last two years at Wellington College in Surrey, between Southampton and London. Returning to America in 1862 he went to Exeter Academy and in 1865, learning that William Barton Rogers was stariting a scientific school in Boston, he did not hesitate a single day in decding to enter the new Massachusetts Institute of Technology. Thus he became and has continued ever since a part of that notable educational institution as student, instructor, Professor of Mining and Metallurgy, and Professor Emeritus. Today, in his ninety-first year he is No. 1 alumnus and holds the unmatched record of seventy years of uninterrupted service in the great cause of engineering education and character building.

Scarcely less notable are the services rendered by Professor Richards to the A.I.M.E., of which he became a member in 1873 at the age of twenty-nine. Always active in its affairs, he was Vice President in 1879–1880, President in 1886, and was elected to Honorary Membership in 1911. He has continued to support at all times and with unflagging zeal the social and technical aspects of the organization over a period of sixty-two years. The records enumerate no less than a score of technical papers published in the Transactions. The first one, read at the Boston meeting in 1873 and published with Volume I of the Transactions, dealt with the Laboratories of Mining and Metallurgy at M.I.T. The last paper deals with Ore Dressing Improvements, read at the Butte meeting in August, 1913. The degree of Doctor of Laws was conferred upon him in 1909 by the University of Missouri, and in 1915 he was awarded the Gold Medal of the Mining and Metallurgical Society of

America for distinguished services in the advancement of the art of ore dressing.

When Richards was a student, there were no laboratories of mining and metallurgy at Massachusetts Institute of Technology or, so far as known, at any other school in the world. Two years later, in the summer of 1870, he joined a party of professors and students, twenty in all, headed by President Runkle, who visited various mining districts in Colorado, Utah, Nevada and California. Suitable machinery for a laboratory was purchased and to Richards was now assigned the task of founding the laboratory and installing the equipment. Accordingly, upon his return to Boston, he went abroad and visited the famous German mining schools at Freiberg and Clausthal and the concentrating mills near by. More than ever, he became convinced of the importance of laboratory equipment for teaching and, in planning the new laboratories in Boston, which were ready for use in 1872, Professor Richards applied new ideas which proved highly successful. He went abroad again in 1876 to visit the tin mines and concentrating mills in Cornwall, the lead districts in North Wales and Scotland, and the lead mines and plants at Mechernich, Germany.

The fundamental idea, which differed from anything that had heretofore been done in laboratory practice, was to combine in the machines and furnaces: (1) size that would match full-scale machines in the quality and character of their work; (2) usefulness, secured by modification of the machines so that the final record showed weights, analyses and a strict account of all products; (3) adaptation to teaching, whereby it was possible to operate the machines under different conditions with changes subject to the will of the student; (4) adaptation to research or testing. A great amount of research has always been done by Professor Richards in these laboratories, not alone because of the service he was able to render the profession, but for the dignity thereby lent to the work of the student.

The outstanding contributions by Professor Richards to engineering literature comprise the Treatise on Ore Dressing (four volumes) and the Textbook of Ore Dressing, both finally completed in 1909. It was in 1893 that R. P. Rothwell, then editor of the *Engineering and Mining Journal*, urged Professor Richards to write a book on ore dressing. Richards thought a year to experiment, a year to visit the plants, and a year to write the book would see the undertaking finished, but he found that there was so much that he did not know, and which nobody else knew, that he began experimenting concurrently with the searching of literature and in 1895 he set out to visit mills. Accompanied by secretaries, he made an extended trip through the mining districts of the Western States. He was wonderfully fitted and equipped by experience to do this work. The development of the experimental laboratories, the early years spent in solving elementary problems, and the later years in fixing the fundamentals of research, prepared him for the work he was now undertaking.

Professor Richards was the first to create the system of mining and metallurgical instruction with the aid of a laboratory, equipped with working machines, wherein the students were taught to execute the lessons of the lecture room; and thus blazed a trail in technical education which other teachers were glad to follow, which now they have converted into a broad, well-marked road. For this, if for nothing else, Professor Richards is entitled to the thanks of the profession.

But in his own specialty—ore dressing, or the mechanical concentration of ores—he has rendered service of equally altruistic character and far broader scope. He has been a developer of principles in an art that was destined to become of premier importance, a codifier of its rules, and the exponent of both theory and practice in a field that was practically uncharted. His monumental treatise on ore dressing was not only the first of such scope in the English language but for many years it was the only one.

The name of Robert Hallowell Richards is known wherever men hoist crude ore out of the mines and dump it into mill bins. He possesses a thousand disciples among the men trained under his guidance and from him they learned not only things mining and metallurgical, to which he gave so much enthusiasm, but guidance of the heart and soul. We salute him for nobility of character, for nobility of service unselfishly rendered, for nobility of attainment in his profession and for nobility of years—may he long continue to enjoy the love and esteem of a host of loyal friends.

W. Spencer Hutchinson.

Cambridge, Mass.
November 27, 1934.

The Hadsel Mill

By R. G. Hall,* Member A.I.M.E.

The method of crushing rock by throwing with violence against a resisting surface does not differ in principle from the method of dropping it from a height upon a specially arranged surface. The former is applied in various mills now in use, especially those for reduction of the softer rocks and minerals. The latter is the principle of the Hadsel mill. Probably not new, but Hadsel's method of approach is at least novel, and applicable to hard as well as softer rocks.

An elevating wheel raises the rock and drops it from a height upon a hard metal plate or series of plates. The first commercial mill consisted of two units, one on each overhung end of a horizontal shaft, driven by a gear train from a 100-hp. motor. A description of this machine, then in operation at a mine at Georgetown, Calif., was printed in the *Engineering and Mining Journal* (April, 1932) and need not be repeated here.

That machine took mine run ores of a size up to 12 in. and successfully produced a pulp of approximately minus 60 mesh, suitable for flotation. The mills were each 24 ft. in diameter and 3 ft. in width of face. The elevating buckets were formed of steel plates, extending inward from the periphery, inclined to the tangent at 75°. This plate was 3 ft., the width of the wheel, and 2 ft. deep. One side of the wheel towards the carrying shaft was closed, the other was open except for the rim plate enclosing the ends of the buckets and extending inward about 5 ft. on the radius.

About a 90° arc was immersed in a concrete tank, which acted as a classifier. The breaking plates, seven in number, were carried cantilever through the open side of the wheel. The lowest plate was placed just above the water level.

The illustrations (Figs. 1 and 2) make plain the general arrangements. The plant set-up was somewhat crude but worked well enough to demonstrate the weak points of the mill.

In this operation it was early apparent that, considering all the factors from delivery of the mine run ore to the final pulp for flotation or cyanidation, the Hadsel mill offered possibilities of great simplicity of plant and savings in power.

Continuous operation developed mechanical weaknesses, some of which were readily discernible in details of design, some became more apparent as operations progressed.

Manuscript received at the office of the Institute Sept. 26, 1934.

* Metallurgical and Chemical Engineer, San Francisco, Calif.

The tank in which the wheel operated proved a poor classifier. This had seemed probable from the beginning. The patentee had made a machine that would crush and grind rock easily and cheaply. Hadsel was not an experienced operator of ore-dressing machinery and soon found out that the only aim of crushing and grinding ores is for the efficient extraction of the contained values. Changes made in the classifier tank were not sufficient remedy. Classifier difficulties arose. The sulfides, released gold particles and coarse sands settled in the bottom of the tank, finally building up to the point where the friction load on the machine became great enough to stop operations. An outside classifier was installed. This improved the quality of the product but failed to cure the main trouble. Friction between the bucket lips and settled

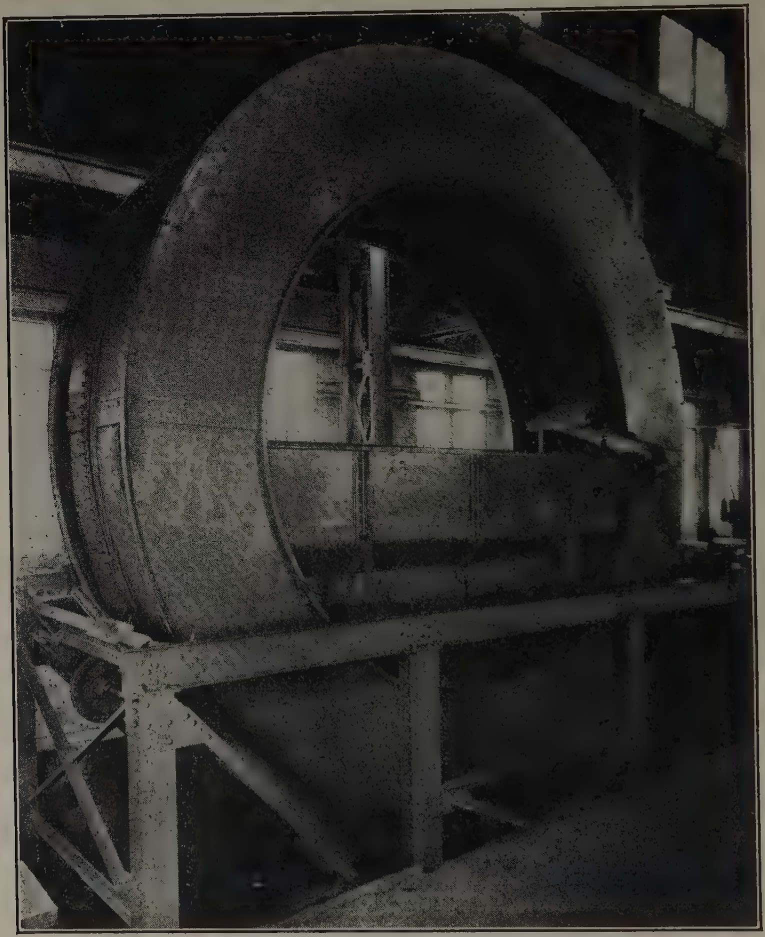

FIG. 1.—A HADSEL MILL.

material in the tank remained inseparable from any design of an open wheel immersed in a tank.

The machine was redesigned. The principles of the sand-wheel elevator and crushing by dropping on plates were retained. The tank or elevator boot was discarded. The mill was mounted on rollers running under steel rails encircling the rim. This discarded the central shaft entirely. The machine was enclosed on the rim and no scoops or openings remained to return material to the grinding circuit. Thus it became as self-contained as a ball mill.

The mill no longer acted as its own classifier but the ground pulp overflowed at one side and entered an outside classifier. The sands returned to the mill. Improved operation and improved products resulted. Power consumption and continuity of operation were both improved, but the delivery of semifinished or classifiable products was unsatisfactory.

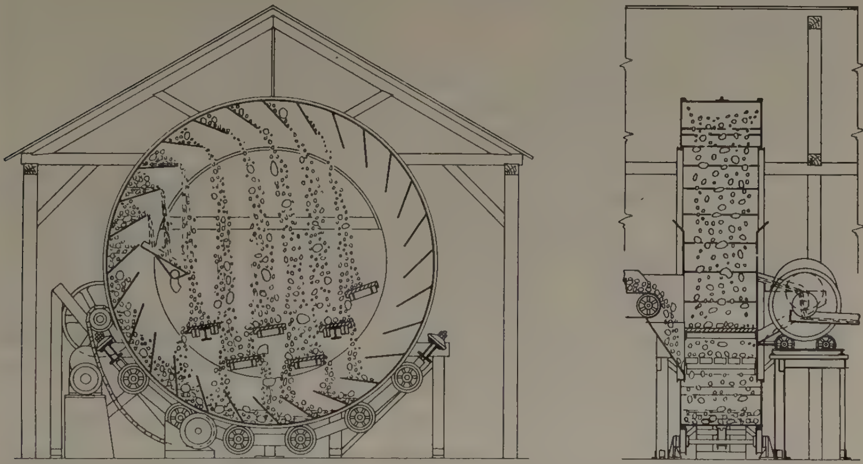

FIG. 2.—PRINCIPLE OF OPERATION OF HARDINGE-HADSEL MILL.

Screens of small mesh, about ⅛ by ¼ in., were paneled into the side plate just inside the lip of the buckets. This prevented unnecessarily large pieces from passing out of the mill with the surges of the water.

Trouble now arose from the accumulation of sands up to 0.5 in. inside the mill, circulating in such a way that they missed entirely passing over the grinding plates. On the up side of the mill, from about 60° from the vertical radius up to about 120°, this accumulation of pulp, sand and water continually flowed down towards the bottom against the filled and rising buckets.

A remedy for this was found in stopping the overflow on the side of the mill. Part way up a deflecting plate, slightly sloped from the horizontal, diverted the pulp from passing back into the bottom. This plate delivers the stream over a screen, the oversize of the screen falls on the grinding plate below and the undersize is diverted outside the mill into a classifier. The vibration of the mill and the action of the circulating water seems to be effective in preventing blinding of the screen. In fact, it is not yet quite certain whether a close-set grizzly may not be as efficacious as a screen, and be stronger to withstand the shocks of occasional larger pieces.

Many attempts have been made to improve the mill. The steps described above mark clearly the advance. As the mill has evolved, it can be made in units of any diameter desired. Probably from 20 to 25 ft., perhaps in extreme cases 30 ft., will be the most efficient sizes.

There are clearly two services to be performed by such a machine as this. In most small and many large mines, the maximum size of rock that comes to the surface will pass a 12-in. grizzly. Such material is proper feed for the Hadsel mill. A slab of unusual size in two dimensions might slip through the breaker plates in such a way as to jam the buckets, but I have not seen such an occurrence. A preliminary breaker will not be needed in most cases, though a grizzly should be provided.

The breaking of these larger pieces and the intermediate sizes down to some size such as one inch is readily accomplished by dropping in a mixed cascade of water and rock from the upper 90° of the wheel on to the plates. This is the impact zone where the rock breaks itself by virtue of its fall

The reduction of the resulting sand and gravel size to a classifiable product is accomplished by the combined impact and attrition of the larger pieces when falling on to the plates and washing these off into the buckets underneath.

It is easy to imagine that a very soft, mushy ore might reduce itself through the first stage, that is, to the sand and gravel stage, very easily, and thereby leave nothing wherewith to finish the job to the finer sizes. I have not seen any example of this.

The other extreme would be a very tough ore where the impact is not sufficient. One might expect such an ore among the quartzose ores of California. One mill takes ores from two mines distant about two miles. With one ore the capacity of a mill 24 ft. in diameter is comfortably over 200 tons daily, while an admixture of the other ore in any considerable proportion brings about an appreciable lessening of output. It seems probable that the ore of the second mine, when used alone, might reduce the finished tonnage by one-quarter or one-third. Such an observation may seem superfluous to those experienced in the operation of ball mills, with their variation in capacity for different ores; but there has been some tendency to believe that if ore A will grind at a certain rate through the mill ore B will do likewise.

It is evident from a study of the principle of the mill that the very largest factor in measuring the crushing capacity is elevating capacity.

The height of drop, that is, the diameter of the mill, must be that necessary to provide impact sufficient to break the largest pieces by virtue of their own falling weight. The design of the buckets and the linear velocity of the discharge lips must be such as to provide proper distribution over the series of breaker plates. Inasmuch as the linear velocity of the bucket lips determines the trajectory for any reasonable variation in wheel diameter, and the linear velocity so far in use is about 200 ft. per minute, it follows that the width of face and depth of bucket determine capacity.

A certain wheel having buckets of 13.5 cu. ft. pitched on the periphery 3 ft. apart has a capacity of 250 tons ground daily. This wheel will have an elevating capacity of $\frac{13.5 \times 200 \times 1440 \times 100}{3 \times 2000} = 64{,}800$ tons per day. In other words, $\frac{64{,}800}{250} = 259$ is the average number of times that rock will be elevated and dropped before being ground.

This figure must not be taken as the circulating load, which is the quantity of material that travels between the grinding machine and the classifier before being fine enough to pass to the next step. So far as one may observe, this classifier circulating load will not differ much from that observed in a well operated ball mill of equivalent capacity. As the classifier operation is a measure of the water requirement, the water used by the Hadsel machine will be the same as that of the ball mill for equal grinding requirements.

In estimating costs of ball-mill grinding, the chief factors are: (1) ball consumption, (2) mill lining, (3) kilowatt-hours per ton, (4) maintenance and repairs, (5) labor attendance. To compare these factors exactly with the Hadsel is not quite possible:

1. The Hadsel mill has no consumption of balls, thereby saving what we may average as about 2.0 lb. per ton ore.

2. Hitherto the Hadsel mill has not been lined at all. The plate-steel construction has not been protected. It is quite evident that the bucket edges, bottoms and sides must be protected. Provision has been made for such lining. What the replacement cost will be is quite impossible to predict. There has been no experience.

Breaker-plate wear comes also under this head. The experience on this point is as yet too little for a definite statement. On one certain mill operating over a period of six months, it would seem that the plates, when made from manganese steel, should exceed three months before replacement. Since the total weight of the plates is just about equal to one pound per ton of the monthly capacity, the cost of replacement without credit for scrap would not exceed one-quarter to one-third pound per ton output.

Taking the two items of wear, the total may approach one-half to three-quarters of a pound per ton output. This total steel consumption would thus approximate one-third to one-fourth of the consumption of the ball mill for equal grinding.

3. Comparisons of power consumptions per ton output can never be exact. The limited experience up to date indicates that for California and Colorado gold mines, units of 100 to 200 tons taking mine run ore should be expected to give a product through 60 mesh with a consumption of 10 to 15 hp.-hr., or, say, 8 to 12 kw.-hr. per ton. This compares favorably with ball-mill consumption for feed of ¾ inch.

TABLE 1.—*Data on Installations of Hardinge-Hadsel Mills*
SUPPLIED BY THE MANUFACTURER

	Beebe Gold Mining Co., Georgetown, Calif.	Slide Mines, Boulder, Colo.	Doss Mining Co., Hornitos, Calif.	Antamok Goldfields, Philippine Islands	Kootenay Belle Gold Mines, Ltd., Salmo, B.C.	Black Bear Mines, Magdalena, N.M.
Size of mill, ft.				24		
Diameter	24	20	20	D 30″	16	20
Width	4½	5	5	old type	5	5
Capacity, tons per 24 hr.	210	55	60	200	50	100
Size of feed, in.	8	12	12	12	10	10
Size of product, mesh	60	28	48	60		
Power used, hp	95	29	40	120		
Installed power, hp	100	40	40	125	40	75
Speed, r.p.m.	2.67	3.0	2.0	2.7	4.25	3.3
Type of discharge	3-mesh screens	Take-off grizzly	Overflow	Overflow	Take-off grizzly	Take-off grizzly
Percentage solids in mill discharge	62	76	20	12		
Mill in closed circuit with classifier						
Size, ft.	5	3 × 8	2	Tank with weir	4 × 10	4 × 10
Type	Esperanza	Hardinge	Esperanza		Hardinge	Hardinge
Total tonnage ground in mill up to Aug. 1, 1934	45,000	7,500	10,000			
Type of ore	⅔ tough silicified schist, ⅓ brittle bull quartz	Vein-mineralized calcite gangue, silicified schist	Heavily mineralized quartz	Tough quartz	To go into operation Oct. 1, 1934	To go into operation about Oct. 1, 1934
Tons per hp-day	2.25	1.9	1.5	1.67		

	Beebe Gold Mining Co.		Slide Mines		Doss Mining Co.		Antamok Goldfields		Kootenay Belle Gold Mines	Black Bear Mines
	Mesh	Per Cent	Mesh	Per Cent	Mesh	Per Cent	Mesh	Per Cent		
Screen analysis of classifier discharge	60	tr.	28	2.1	−200	55	60	0.5		
	80	0.7	35	3.6			100	7.0		
	100	3.2	48	9.3			200	7.0		
	200	24.2	65	8.0			−200	63.5		
	−200	71.7	100	12.2						
			200	12.0						
			−200	49.9						

TABLE 2.—*Specifications of Hardinge-Hadsel Mills*

SUPPLIED BY THE MANUFACTURER

Mill Size, Ft.	16×3	16×3	16×4	16×5	20×48	20×4	20×5	20×6	20×8	24×5	24×6	24×8	24×10	30×8	30×10	30×12
Capacity, tons per 24 hr....	25	35	45	60	70	110	140	170	225	275	330	450	560	700	900	1,050
Size Hardinge classifier used, ft.	3×8	3×8	3×8	3×8	3×8	4×10	4×10	5×12	5×12	6×14	6×14	7×16	8×20	8×20	9×24	10×28
Weight mill (no classifier), lb.	25,000	29,000	33,000	38,000	48,000	54,000	60,000	67,000	78,000	95,000	110,000	135,000	160,000	220,000	270,000	325,000
Weight mill and classifier, lb.	28,000	32,000	36,000	41,000	51,000	61,000	67,000	79,000	90,000	114,000	129,000	160,000	200,000	260,000	325,000	400,000
Motor required, hp.	20	30	40	50	50	75	100	125	150	200	250	300	400	450	600	700
Countershaft, r.p.m.	220	220	220	220	198	198	198	198	198	190	190	190	190	150	150	150
Maximum feed size through sq. hole, in.	10	10	10	10	10	10	10	10	10	12	12	12	12	15	15	15
Diameter of mill, ft.	16	16	16	16	20	20	20	20	20	24	24	24	24	30	30	30
Width of mill, ft.	3	3	4	5	4	4	5	6	8	5	6	8	10	8	10	12
Bucket depth, in.	14	16	16	16	18	24	24	24	24	30	30	30	30	36	36	36
Bucket load, lb. (at 120 lb. per cu. ft.)	240	340	430	540	600	950	1,200	1,440	1,900	2,040	2,400	3,200	4,000	4,800	6,000	7,200
Number buckets	28	28	28	28	28	28	28	28	28	32	32	32	32	32	32	32
Mill r.p.m.	4.25	4.25	4.25	4.25	3.30	3.30	3.30	3.30	3.30	2.85	2.85	2.85	2.85	2.25	2.25	2.25
Pounds per minute dropped	28,500	40,000	50,000	67,000	56,000	85,000	110,000	130,000	180,000	185,000	220,000	300,000	363,000	345,000	435,000	520,000
Tons per 24 hr. dropped....	20,500	29,000	36,000	48,000	40,000	61,000	79,000	95,000	130,000	135,000	160,000	215,000	263,000	250,000	315,000	375,000
Drops at rated capacity to grind	820	830	800	800	570	560	560	560	580	483	483	475	472	356	350	356
Horsepower to lift rated load (theoretical)	13	18	22	30	30	46	60	70	97	118	140	190	232	275	350	420

On the credit side of the Hadsel is to be placed the almost certain elimination of coarse breakers on all but the largest installations, and the certain elimination of all intermediate crushers. The resulting savings in installation and operating costs are a decided factor in favor of the Hadsel principle.

4. During the development period experience in the item of maintenance has been decidedly against the Hadsel mill. The earlier units incorporated errors in design and weakness in construction, which in some cases entailed losses of time and expensive changes far outweighing any advantages. It is fair to say that these troubles have existed in an acute form only in certain older installations. Even in these cases they have been contributed in no small degree by the ignorance and incompetence of some of the operators.

5. Operating labor and maintenance, considering the elimination of coarse and intermediate crushers, should show an advantage in favor of the Hadsel principle.

The accompanying data sheets (Tables 1 and 2) have been contributed by the manufacturers. I have not personally checked up all the information contained therein.

CONCLUSIONS

The principle of the Hardinge-Hadsel mill has been demonstrated as sound. The machine will take ordinary mine run ore and will reduce it to flotation or cyaniding size in one operation when working in closed circuit with a classifier. This much is established.

It is also demonstrated that in doing this there will be certain economic advantages: (1) simplicity of crushing and grinding plant, accompanied by a saving in installation cost; (2) saving in power consumption with improvement in load factor; (3) saving in consumption of grinding steel.

Accompanying these undoubted advantages realized from the application of the principles of the machine, there have been development difficulties of a serious nature. Some errors, perhaps the most serious, have resulted from lack of experience, inherent in all machinery departing from a tried and proved type. The most obvious have been corrected. No doubt there will arise in each installation others that will require patience and ingenuity to overcome. Maintenance and repairs, at least in the earliest installations, remain too high, though I understand these are being reduced to a reasonable amount in later models. Continuity of operation, as in a well designed ball mill, has not been attained entirely so far as I know, but I can see no reason why such continuity should not be entirely possible.

In passing judgment on such an important development, it will be well to bear in mind that the machine, even in the earliest models, has demonstrated the ability both to crush and to grind rock by the application of gravity of the falling material. Economies in power and steel consumption have been demonstrated.

It is true that weaknesses of design and construction, some of them quite obvious and unnecessary, have developed. These have been corrected. I see no reason why a design and construction should not be attained that will give the same reliability and continuity of operation, and the same low cost of maintenance and repairs as we now expect—and receive—in a good ball mill.

Judging by the progress already made, and the results to date, the most conservative operator may well be encouraged to believe that the Hadsel mill marks a distinct forward step in machinery for ore reduction.

Ball Milling*

By Alexander M. Gow,† M. Guggenheim,‡ Junior Member A. I. M. E., A. B. Campbell,‡ and Will H. Coghill,§ Member A. I. M. E.,

(New York Meeting, February, 1934)

The object of this paper is to discuss the fundamental principles of ball milling and to present some observations which have been made in laboratory and plant investigations. The discussion will be limited to wet grinding of ores in cylindrical mills, but doubtless many of the principles involved will be equally applicable in the other fields of fine grinding in which mills of the same type are used.

In the concentrators of the United States there are about 650 ball, rod, or pebble mills, operating or ready to operate, and these have about 100,000 connected horsepower. For grinding in cement plants there is about three times this connected horsepower, and other industries have an unestimated amount, all used for the general type of grinding that is to be discussed here. It is true that these figures do not represent stupendous amounts of power, but when one considers that ball milling is often the "bottle neck" in a concentrator and that it often accounts for the largest single item in the cost sheet, the importance of a comprehensive understanding of the principles of ball milling can be appreciated.

Much of the past literature devoted to the subject has been concerned with the Kick-Rittinger dispute over evaluating grinding, or with dissertations on ball paths and the closely allied impact versus attrition hypotheses, or with the exclusive consideration of certain specific variables, such as mill speed or circulating load, irrespective of the other factors in operation. Such subjects may be dismissed with but little discussion in what may be called, by comparison, a new philosophy in the study of ball milling. Briefly, this "new philosophy" consists of using power as an index of the mill operation, and investigating grinding from the standpoint of set and induced variables, with emphasis on the latter.

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References to the literature will be made only when they will aid in preventing unnecessary repetition.

Evaluating Capacity and Efficiency

In calculating the relative amounts of grinding the Rittinger law of reciprocals has been accepted, and surface calculations have been made, using the Tyler $\sqrt{2}$ screens. For convenience, and since the values are only relative, the material retained on the 1.05-in. screen was assigned a value of one surface unit per unit weight. The values for the finer screens were made reciprocally proportional to the relative sizes of the openings; Table 15 shows an example. Grinding *capacities* are evaluated as surface tons per hour and grinding *efficiencies* as surface tons per net horsepower-hour. All calculations are made in terms of useful grinding; that is, the mill is given credit for grinding only to a specified or limiting size, not beyond. In the notes that follow these values of capacity and efficiency will be expressed with the limiting sieve size in parenthesis.

Ball Paths and Trajectories

Ball paths, particularly the trajectory of the outermost layer of balls, were once looked upon as offering a key to the analysis of ball milling. At one time it was thought that the balls followed parabolic paths like projectiles, but by use of a mill with screen ends the authors demonstrated and reported in a paper:[1]

> . . . That the balls do not act independently when they leave the shell, but continue in contact, pushing those ahead until they pass the apex of their flight. The continuous upward stream of balls cannot lose velocity and consequently there is a horizontal velocity at the apex of their flight equal to the peripheral velocity of their circular course. From this point they are free falling except as they are hindered by atmospheric friction, crowding, and impact.

To illustrate further the effect of *pushing*, an inclined glass tub bent upward at the lower end was used. Balls that were dropped througe the tube were shot upward from the lower end and:

> Showed that when a succession of several balls is projected upward, the first of the series goes farther than a single ball shot at the same angle and velocity.

Adverse criticism was made of that paper because screen ends were used on the ball mill, but the screens served their purpose in permitting observation of what should have been recognized from the beginning—that the undisturbed path in a ball mill is not that of the proverbial cannon ball. The criticism was of the tool, not of the observations. Now, the authors have a confession to make—not that they erred in the methods or conclusions of the investigation of ball trajectories but that they spent time on a subject which, as will be shown by this paper, has little to do with the common-sense study of ball milling.

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[1] A. M. Gow, A. B. Campbell and W. H. Coghill: A Laboratory Investigation of Ball Milling. *Trans.* A. I. M. E. (1930) **87**, 51.

Impact and Attrition

Ball-path observations have led quite naturally to the idea that high, "cataracting" speeds, which caused the balls to be thrown through the upper region of the mill, resulted in grinding by impact as the balls hit the shell and each other, whereas slow, "cascading" speeds, with the ball-mass rolling or sliding in the mill, ground by attrition. The idea is still held that these are distinct types of grinding; however, comparative tests have shown that the difference between slow-speed grinding (supposedly attrition) and high-speed grinding (supposedly impact) is one of degree rather than kind. As will be shown in the discussion of tests, the amount of grinding, provided the operating conditions are what might be called "normal," is proportional to the power expended. If grinding at 30 per cent of the critical speed[2] has seemed to differ from that at 80 per cent the difference should be attributed to the power expenditure and the effect of the induced variables rather than to the difference between attrition and impact grinding. For example, when the speed of an operating mill, running at about 65 per cent of the critical, is increased and the feed rate is kept the same the ground product becomes finer and contains less of the coarse sizes. This additional grinding, particularly of the coarse particles, is sometimes attributed to the additional impact, but in reality it is the result of more power doing more grinding of the same kind. If the feed rate had been increased in proportion to the rise in net power the grain size of the discharge would have been about the same; but the increase of the feed rate would have caused some other changes also, which would have complicated the analysis had *speed* alone been the subject under observation.

Variables, Set and Induced

The present investigation has shown that the variable factors in ball milling must be divided into two groups, the set variables and the induced ones, and that the interrelation of these must be understood before ball milling can be anything but a cut-and-try operation. For example, the amount of ore pulp in the mill at any instant is an important factor in the grinding; this is acknowledged in batch grinding, when the best charge is predetermined and used. In continuous grinding this is one of the induced variables. It depends on the feed rate, the size of the discharge opening, the ball charge, and other set variables. The influence the set variables exert on the induced ones and the effect of both on the power and grinding will be discussed in detail.

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[2] Critical speed is the speed at which the outer layer of balls would centrifuge if they did not slip.

Field Contacts

In this investigation the facilities for studying ball milling have been unusually favorable. Two large mining districts have been accessible, and the operators have cooperated heartily in the correlation of laboratory and plant testing. One district has about 50 ball and rod mills and the other about 30. For the investigation in plants, a variety of sizes of pulleys and pinions was made available to permit systematic changes in speed. When it was desirable to convert a rod mill to a ball mill and again back to a rod mill this was done. One mill was dumped and packed to determine dead load. In other tests ball charges were varied to suit conditions, and grinding media were changed to investigate the effect of sizes. Feed rates of 1 to 100 tons per hour have been observed, and recording power-meter charts have been obtained during much of the field testing. One of the plants in which testing was done is shown in Fig. 1.

Fig. 1 —A crushing plant, sampling plant and concentrator in the Tri-State district, Picher, Oklahoma.

The type of power chart obtained from various mills has been of passing interest. In one instance a power bobble of 20 per cent was recorded as the scoop of the feeder dug into the sand in the feed box; on another there was a jump as the splice in the belt slapped over the motor pulley; while a third, a large, direct-connected rod mill converted to a ball mill, gave a smooth, steady line on the power chart.

In the laboratory, mills of many sizes have been employed, but tests showed that a 2-ft. mill could be used to forecast the work of a commercial mill. The laboratory tests with which this paper deals were made with mills of the following sizes: 1½ by 2 ft., 2 by 2 ft., and 2½ by 2 ft. These mills were driven through rollers so that the heads would be unencumbered and accessible to changes. Electrical power measurements were made so that the net power expended within the mill could be calculated. One closed-circuit set-up is shown in Fig. 2.

In this particular flow sheet the discharge from the ball mill (1) ran into the intermittent raking classifier (2). Oversize or circulating load was returned by the inclined belt (3) and dumped on to the weightometer (4) which returned the oversize to the ball mill. New feed was delivered to the ball mill by the feeder (5), which was controlled automatically by the weightometer. When the circulating load was too heavy the weightometer shut off the motor on the feeder, and when the circulating load was too small the feeder was turned on automatically. By setting the beam on the weightometer, a circulating load of any amount could be maintained. The classifier was designed and developed by the authors in connection with the investigation. It has been described elsewhere.[3]

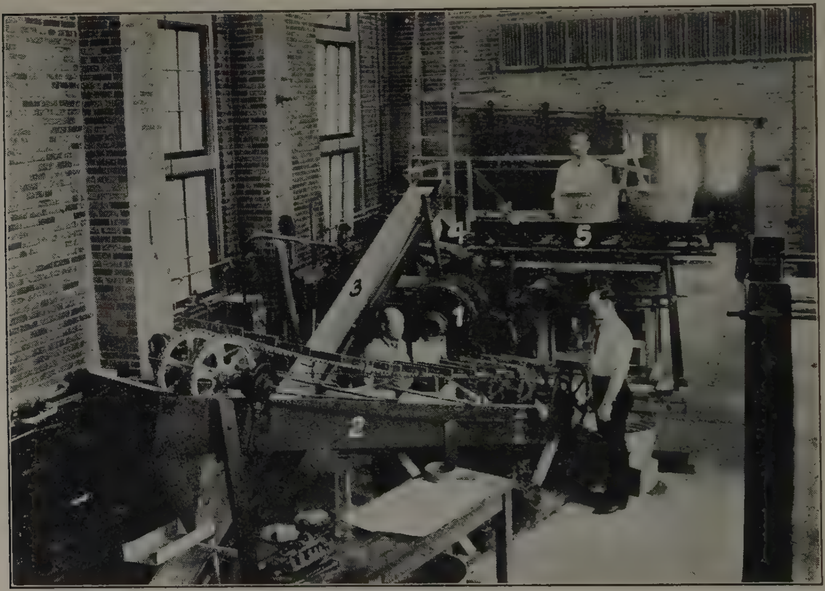

FIG. 2.—ONE OF THE LABORATORY SET-UPS FOR CLOSED-CIRCUIT GRINDING WITH A 2 BY 2-FT. BALL MILL.

A good mechanic built practically all the laboratory equipment.

Feed for laboratory grinding was obtained from the field in carload lots. Extremes in hardness were found in Tri-State flint and Southeast Missouri dolomite. The former was tailings from a jig feed that had been crushed through 1/8-in. by rolls in closed circuit with screens. The dolomite sample was cut from the tailings of a selected concentrating table so that its grain size was the same as that of the flint. Both samples were practically one-mineral products and homogeneous. The similarity of grain size facilitated comparative grinding tests.

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[3] Intermittent Raking Drag Classifier. *Rock Products* (Dec. 10, 1932) **35**, 25.

Power Studies

Power is often the largest item in the cost of commercial operations. It may be considered the most enlightening single factor in the study of ball milling, and its study offers a solution to the problem of cutting operating costs. Power is used to impart action to the grinding media, and the amount of grinding varies, within limits, as the ball action. In ball milling it is not the grinding but the ball action that consumes the power. The authors believe that scientists have yet to show that ground rock contains appreciably more energy than unground rock. The kinetic energy expended in ball action is transferred chiefly into heat. An old scientist spoke wisely when he said that heat is the boneyard of all energy. When conditions are suitable fracture may accompany the energy transformation; when those conditions are uniformly suitable grinding becomes proportional to the power expended.

These power studies will take up, first, the effect of the different variables on the power, and second, estimation of the power required by commercial mills under various conditions.

Dead Load

In commercial operation the gross power input to the ball-mill unit is the quantity of economic interest to the operator, but from the scientific standpoint the study must be made of the net power expended within the shell. The difference in these two values is motor inefficiency and the dead load.

To determine the dead load of a commercial mill is a laborious task. The entire ball charge must be dumped from the mill, and then the equivalent weight must be mounted centrally within the shell so that the mill becomes as a flywheel of equal gross weight. The power to rotate this set-up constitutes the dead load. Such a test was made of a commercial belt-driven mill,[4] and the dead load was found to be approximately 15 per cent of the gross power input. The dead load, being primarily the result of friction, may be considered directly proportional to the speed; hence if one determination is made the intermediate values may be calculated for tests at various speeds. The motor loss was 10 per cent, therefore the net power was approximately 75 per cent of the gross power input.

In the laboratory study, only net powers were considered, since the dead load and motor loss were characteristic only of the particular set-up.

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[4] A. M. Gow and M. Guggenheim: Dead-load Ball-mill Power Consumption. *Eng. & Min. Jnl.* (1932) **133**, 632.

Power and Operating Variables

Many operating variables affect the power, and since all cannot be considered simultaneously the more important will be considered first to form a basis for discussion and to furnish power curves, which may be subject to slight correction due to variables of less importance.

Speed and Ball Charge

First, the effects of the *speed* and of the *amount of the ball charge* will be considered. Fig. 3 presents net power curves for the 2 by 2-ft. laboratory mill run at various speeds and with ball charges of various volumes,[5] as shown. In this case, the feed and discharge openings were both 4 in. in diameter, and the feed was dolomite fed with water at a slow rate of about 5 lb. per minute. Balls were mixed, 1½ to ¾-in., and the mill shell had twelve ⅜-in. lifter bars to simulate a wave lining.

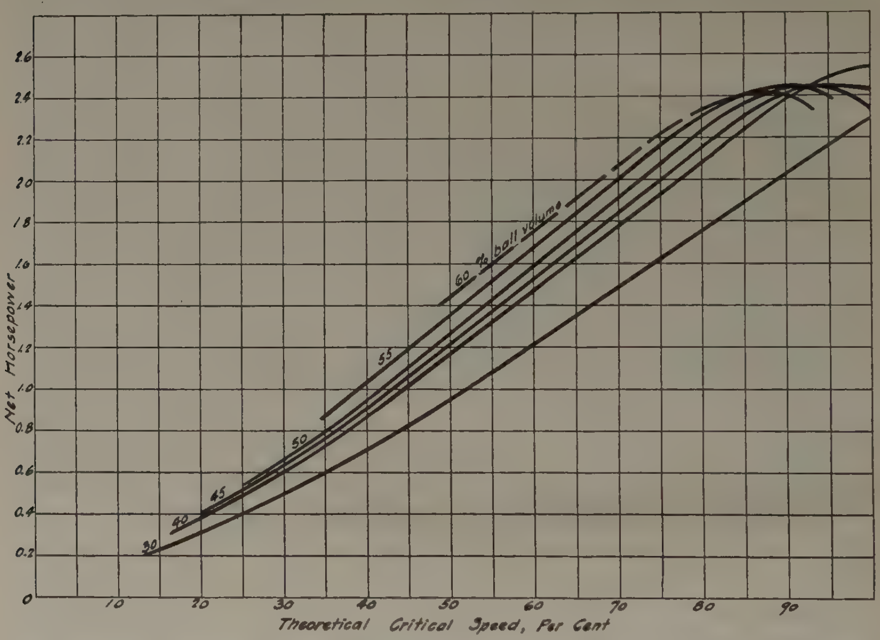

Fig. 3.—Basic power values at various speeds and ball volumes, 2 by 2-ft. ball mill.

Although the power for 60 per cent ball volume lies above the 55 per cent curve, this probably never occurs in practice; loading above 55 per cent generally causes a reduction in power, as shown later by the observed values in Table 1. Therefore, loadings of 55 to 60 per cent ball volume should use the 55 per cent values. (See example 6 of the power estimates.)

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[5] Ball volumes are expressed as percentage of the total mill volume occupied by the balls when at rest and including interstices.

From the values on the curves in Fig. 3 the power required to grind in commercial mills under various conditions may be estimated, but other factors should be considered before such estimates are as nearly correct as they might be. Therefore, before the forecasting of actual powers is discussed, the corrections, if they may be so called, will be considered. It should be noted that the curves break slowly at high speed, in contrast with the sudden drop observed by other experimenters in tests using small laboratory mills containing a few large balls. The 2-ft. mill is large enough to simulate a commercial mill.

SIZE OF DISCHARGE OPENING

The size of the discharge opening affects the power; that is, the curves in Fig. 3 may be raised or lowered as the discharge opening is increased or decreased. The reason for this is found in the ball action, as it is affected by the amount of pulp in the mill. A small opening retards discharge and holds pulp in the mill. The large amount of pulp swells the mass of material within the mill, moving the charge toward the axis where less power is consumed. A larger opening reverses the situation, and the power goes up.

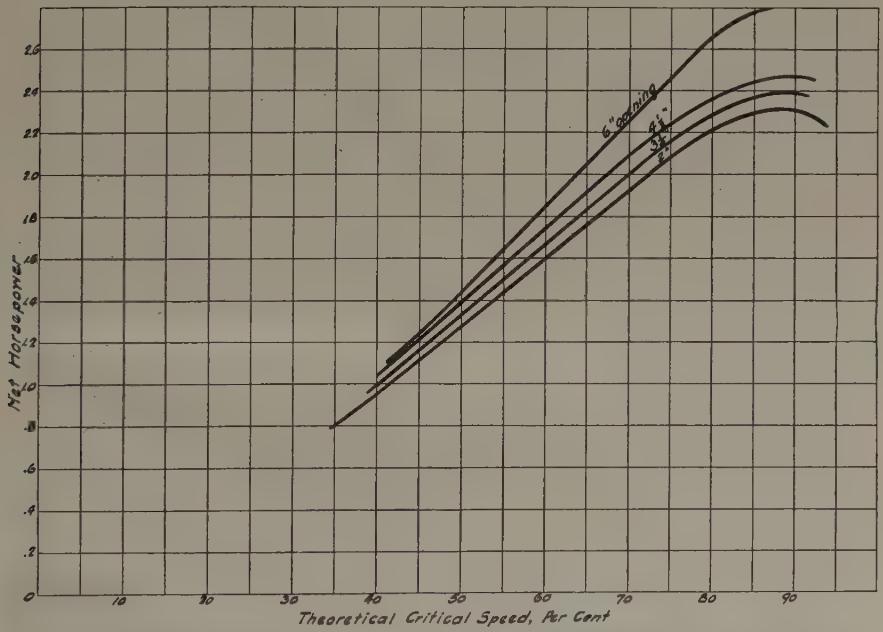

FIG. 4.—POWER VALUES AT VARIOUS SPEEDS FOR DIFFERENT SIZES OF DISCHARGE OPENING.

A series of tests was made with the laboratory mill, in which all conditions remained the same except the size of the discharge opening. The powers varied as shown in Fig. 4, which gives power curves for discharge openings of 2, 3¼, 4½ and 6 inches.

FEED RATE

The feed rate affects the power in the same manner as does the size of discharge opening; a small feed rate is equivalent to a large discharge opening, and a large feed rate causes swelling of the mill's contents, as does a small opening. However, the total influence of feed rate is not quite so great under reasonable operating conditions. An example of this change is shown in Fig. 5, which gives the power used, as flint and as dolomite were ground under conditions that were similar, except for feed rate. The dolomite, being softer, was fed at a more rapid rate than the flint.

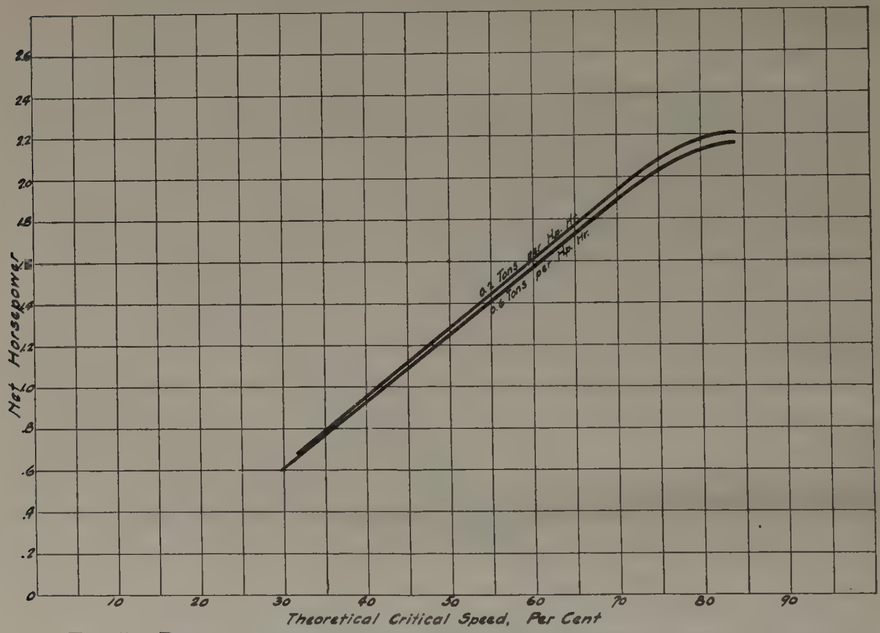

FIG. 5.—POWER VALUES AT VARIOUS SPEEDS FOR DIFFERENT RATES OF FEED.

The length of the mill is a similar factor when the mill is so long that the flow of pulp is retarded by a small gradient. Small openings, large feed rates and long mills all tend to increase the amount of pulp within a mill and to reduce power.

Other factors, such as the specific gravity of the ore and the pulp density, no doubt have less effect on the power and may be disregarded for the present. They will be discussed under pulp density.

SLIPPING

Slipping confuses speed estimates and changes power from its normal values. In general, a charge that slips consumes the same power as it would have used at a lower speed if it had not slipped, but at high speeds there are exceptions even to this rule. The power curves given thus far lie within the field of the most common practice, but their scope is not sufficient to explain all possible conditions.

Power is made up of two components: (1) the lifting of the balls, permitting those in the outer layer to return to the bottom of the heap while the inner layers are in a state of continual adjustment and relative motion, and (2) slipping (or slipping and rolling) of the balls and ore on the breast of the mill. Either component may vary from zero, nearly to the exclusion of the other. The curves given so far are from tests in which slipping was minimized by the use of lifters against which the balls and ore were keyed. This keying did not combat the relative motion completely, as shown by the fact that maximum power—the power at that speed above which the balls are thrown so high that they give back some of the energy expended—came at about 85 per cent of the critical, whereas in a short mill with heads only 6 in. apart,[6] and equipped with lifters, the balls had been keyed so thoroughly and slipping had been reduced so effectively that maximum power came at even slower speeds.

In the experiments to be considered now slipping was allowed to run the course more freely than in the usual practice. The breast of the mill was cast as a unit and was without lifters or liners, therefore it was smoother than that of a commercial mill because, although the liners might be considered smooth, the joints between the liners of a commercial mill act as lifters.

The following series of tests to investigate slippage was made in a 2 by 2-ft. batch mill:

1. Power as various charges of dusted and nondusted balls were tumbled at various speeds.
2. Power at various speeds as ore and water were fed into the mill in small increments.
3. Power at various speeds as coarse ore was ground to pulverized ore.

1. *Power for Tumbling Balls*

After prolonged tumbling by themselves cast-iron balls became exceedingly smooth, as though covered with graphite. Because the smoothness made a great difference in the power and amount of slippage two sets of tests were made, one in which slipperiness was permitted and one in which a handful of sand was added to cut the gloss and dust the surfaces.

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[6] A. M. Gow, A. B. Campbell and W. H. Coghill: Reference of footnote 1, 70.

Ball volumes of 30, 40, 50 and 55 per cent were used, and the power readings were taken at shell speeds 40 to 100 per cent of the critical, or above. Thus, four tests were made for each condition of balls. The results are shown on the curves in Fig. 6.

The first pair of curves (30 per cent ball volume) show the characteristic slippage relationship between power and speed. With this light load of balls the shell slipped underneath the charge in a manner analogous to that of a muller on a bucking board; the faster the muller is dragged over the board, the greater the power consumption. The balls slipped on the shell so much that the shell speed could be carried above the theoretical critical without a break in the power curve. The nondusted balls slipped more than the dusted ones, as shown by the greater speed of the shell necessary to give the same power consumption. Only enough sand was added to place a coating on the balls, and yet it increased the required power about 12 per cent. In both tests the power varied directly with the speed, and the curves are straight lines running approximately through zero.

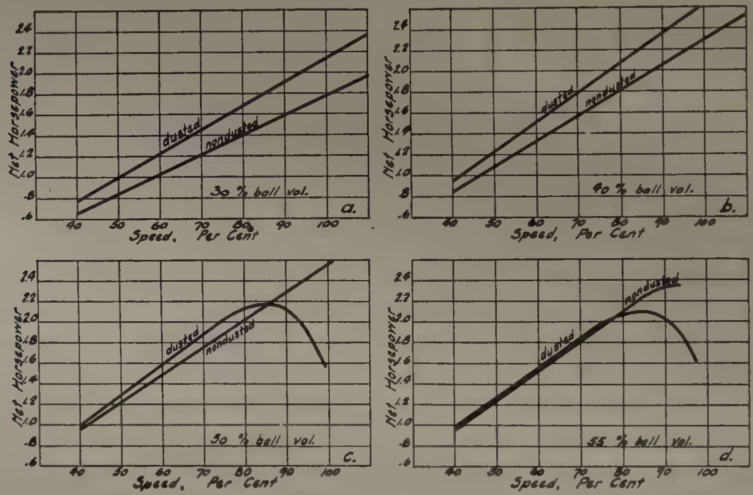

FIG. 6.—POWER FOR TUMBLING DUSTED AND NONDUSTED BALLS AT VARIOUS SPEEDS AND BALL VOLUMES.

The next curves, those with 40 per cent ball volume, are of the same general type—extreme slippage. The increased load consumed more power and made the lines steeper. The values for the dusted and nondusted balls, as the ball volume was increased, lay successively closer together, indicating that the surface condition is of relatively less importance and that there is less slippage to be combated by keying as the ball volume is increased.

Half filling the mill reduced slipping so much that the dusted balls showed a power slump at the high cataracting speeds. The nondusted balls continued to slip, but the amount was reduced, as shown by the proximity of the two curves, and in all probability they, too, could have been centrifugalized if the speed had been carried up to about 140 per cent of the critical.

Loading the mill to 55 per cent of the volume gave a condition that nearly eliminated slipping among the dusted balls and reduced it materially with the smooth balls. At this loading the maximum power for the dusted balls did not reach as high a value as it did for the 50 per cent ball volume. Evidently the overloaded conditions tended to balance the force required to lift the balls. The greatest difference between the curve is in the high-speed range. High cataracting caused a rapid drop in power with the dusted balls. Such a decrease was impossible with the non-dusted balls because when part of the balls went into the air the remaining volume was so small that slipping was induced; the ball action could increase only to a certain point, beyond which additional slipping made up for increases in speed. The ball action was indicated by the power study and also checked by observations through a screen-end on the mill.

The high power attained with a slipping charge at high speed suggests grinding under these conditions when high capacity is desired; however, the feasibility of this procedure is questioned when one considers the consequences of centrifugal action if some condition were suddenly to reduce the slipping.

2. *Power on Gradual Addition of Ore*

Some mention has been made of the effect on power exerted by the amount of pulp retained on a continuously operating mill. Another phase of this same subject bears on the amount of slipping. The effect of small amounts of pulp was investigated by starting with balls alone and intermittently adding small parcels of pulp until it backed out through the feed trunnion. This was done at several ball volumes and at various pulp densities. Fig. 7 presents diagrams showing the power consumption at various speeds and ball volumes as the amount of pulp was increased in a smooth mill. The pulp density was 67 per cent of solids by weight.

In the first two diagrams—those for ball volumes of 32 and 42 per cent, respectively—the curves for the different speeds are spaced evenly; the power is proportional to the speed even up to the critical. As pointed out in Fig. 6, this indicates extreme slipping. In both these cases and at all speeds the power rose as ore and water were first added to fill the mill. The maximum power was attained when the ore was approximately sufficient to fill the interstices of the balls at rest. The larger volume of balls required slightly more ore, and so did the higher speeds. The final power after the mill was filled was less than that required when there was no ore in the mill.

The last two diagrams in Fig. 7 show that when the ball volume was large slipping was affected by the amount of pulp in the mill. By referring back to a power-speed curve of little or no slipping, such as Fig. 6*d* dusted, one can see that the amount by which the power for 100 per cent speed is less than the power for 80 per cent speed indicates the degree to which slipping has been reduced. The greater the reduction in slipping, the smaller is the relative value for the power at the critical speed; therefore, a power curve made at the critical speed is low when slipping is small in amount and high when there is much slipping.

With a ball volume of 48 per cent and high speed the addition of only 20 lb. of ore reduced slipping, but further additions were associated with increased slipping. The variations in the amount of slipping causes, and is indicated by, the irregular power curves at 90 and 100 per cent speeds. The same conditions hold at a ball volume of 53 per cent where additional balls also tended to reduce slipping.

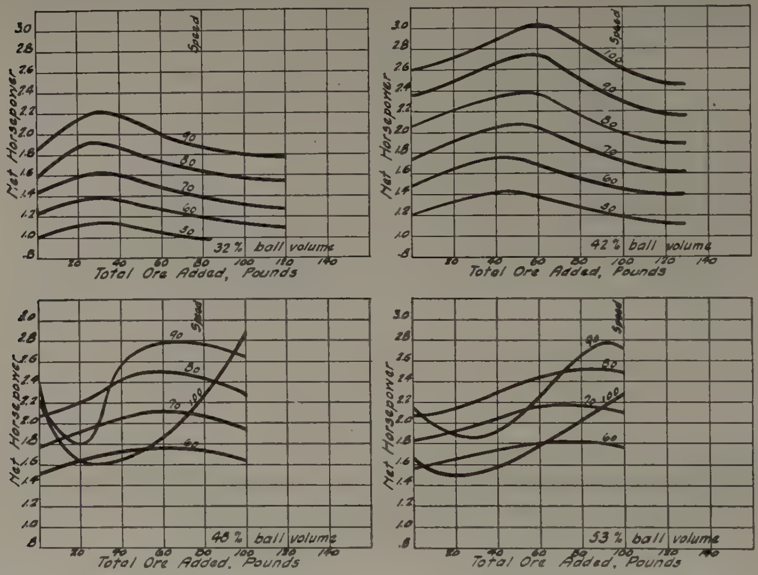

FIG. 7.—POWER AT VARIOUS SPEEDS AND BALL VOLUMES AS PULP OF 67 PER CENT SOLIDS WAS ADDED GRADUALLY.

The irregularity of the power for speeds of about 80 per cent warns operators of the hazards of running a mill at very high speeds.

The power upon gradual addition of ore was also studied at various pulp densities. The ball volume remained at 42 per cent, and the pulp density of the additions was set at 67, 75 and 80 per cent of solids, respectively. Data from these runs are shown in Fig. 8. With the thicker pulps larger amounts of ore were required to reach the point of maximum power, and the maximum powers themselves were higher. Thickening the pulp had much the same effect in reducing the slipping as did increasing the ball volume in the previous series. Again, the irregularity at high speeds forecasts difficulties in operating if these are to be used in practice.

In both these series of tests the ore added near the start of the test became ground exceedingly fine; probably some of the slipping near the end of the run was attributable to the fineness of the ore. This led to the next group of tests.

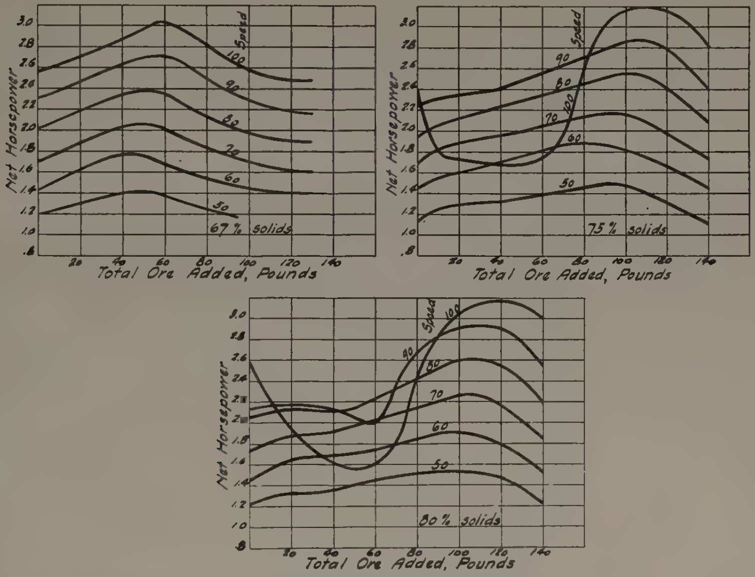

FIG. 8.—POWER AT VARIOUS SPEEDS AND PULP DENSITIES AS PULP WAS ADDED GRADUALLY; 42 PER CENT BALL VOLUME.

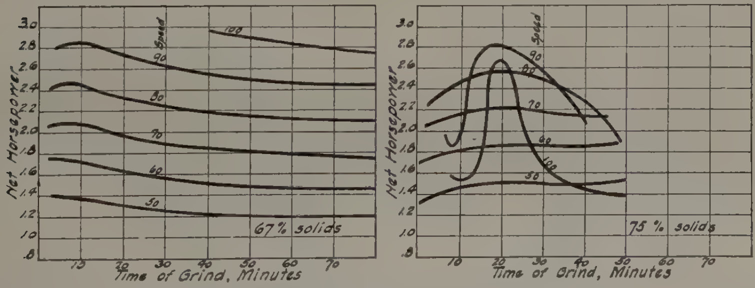

FIG. 9.—POWER AT VARIOUS SPEEDS AND PULP DENSITIES AS 100 LB. OF COARSE GRAVEL WAS GROUND TO FINES; 42 PER CENT BALL VOLUME.

3. *Power While Coarse Ore Was Being Ground Extremely Fine*

To find the effect of grain size the mill was charged with balls, water and gravel, and power was observed while the grinding reduced the grain size. Two tests were run with a ball volume of 42 per cent, 100 lb. of gravel, and water to give pulp densities of 67 and 75 per cent solids, respectively. The results are shown in Fig. 9. As grinding progressed the pulp became either a slime or a thick mud, depending on the amount of water. A slime tended to lubricate the balls and thereby aid slippage, whereas, thick mud caused the balls to adhere and in extreme cases to be plastered to the shell and to each other. In either instance, the slippage could not be hindered by coarse particles because these were ground out. The effective viscosity or bonding power of the pulp had much to do with the type of ball action. The tests show that slipping, with its resulting effect on power, may be increased or diminished by the size and distribution of grains and by the pulp density.

The results of these slippage tests have drawn attention to factors that affect power consumption. If operating conditions are as variable from mill to mill, or from plant to plant, in commercial practice as they have been in this part of the investigation it is little wonder that manufacturers and operators do not rely more on technical data to estimate the power required for their mills. Although the examples just cited represent extremes it is believed that their parallels have occurred with enough frequency to cause confusion. However, the extreme cases, while important in a study of principles and occasionally met in practice need not confuse the study of what may be considered normal operation.

Power for Various Mills

The next step in applying the power study to commercial mills is to see how the size of the mill affects the power and how mills of various sizes may be compared. The power in imparting action to the balls is used in lifting a certain weight of balls on a lever arm that is proportional to the mill's radius, and at a speed proportional to the revolutions per minute of the shell. Since the weight of balls in a unit of length of mill is a function of the square of the radius, because the lever arm is directly proportional to the radius, and since the critical speed, or any percentage thereof, is an inverse function of the square root of the radius, the horsepower is a function of the radius raised to the 2.5 power.[7] Thus:

$$\text{Horsepower} = Ar^2 \times Br \times Cr^{-0.5} = Hr^{2.5} \qquad [1]$$

where

A is a constant for the weight of the balls per unit volume,
B is a constant for the lever arm,
C is a constant for the critical speed,
H is a constant for horsepower.

This theoretically derived exponent may be used without modification in comparing two mills only when operating conditions are identical.

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[7] Acknowledgment is due to Mr. H. B. duPont, of the E. I. duPont de Nemorus Co., for this simplified analysis.

Such a condition rarely exists in practice on account of slipping and other induced variables. Empirically, the exponential value was found to be 2.6 when mills only 6 in. long were used.[8] Other tumbling tests using mills 2 ft. long and 18, 24 and 30 in. in diameter gave an empirical value of less than 2.5. It is possible that in mills of greater length the value might appear to be even less, not because the value has actually changed but because the quantity of slipping made it more difficult to estimate accurately the mean speed of the ball mass.

FORMULA FOR ESTIMATING POWER

In looking into a ball mill one can observe that the central portion—midway between the ends—does not have as much ball action as do the sections near the ends. As a result of this variation throughout the length of a mill the power should not be considered directly proportional to the length, particularly in comparing long mills with very short ones. The following empirical formula, hypothecating the mill as divided into two or more 2-ft. sections, one of which has the ends and the others are without ends, is offered as furnishing a reasonable estimate of the power required to run a cylindrical ball mill:

$$\text{Net horsepower} = \left[\left(\frac{L}{2} - 1\right)K + 1\right]\left(\frac{D}{2}\right)^{2.5} \times P \qquad [2]$$

where

L is the inside length of the mill in feet,

K is 0.9 for mills less than 5 ft. long; 0.85 for mills over 5 ft. long,

D is the mean inside diameter of the mill in feet,

P is the net power used by the 2 by 2 ft. laboratory mill under similar operating conditions.

FIELD EXAMPLES OF USE OF FORMULA

Example 1.—A 6 by 4-ft. ball mill is loaded to 50 per cent mill volume with balls and run at 80 per cent of the critical speed. The discharge opening is 9 in. and the feed rate 15 tons of dolomitic ore per hour. Actual inside dimensions of the mill are 5.8 by 4.2 feet.

Since the opening, the feed rate, and the specific gravity of the ore are reasonably similar to those used in the laboratory mill, the value P may be taken directly from Fig. 3. At 50 per cent mill volume and 80 per cent speed P is 2.23 hp. Substituting in formula 2:

$$\left[\left(\frac{4.2}{2} - 1\right) \times 0.9 + 1\right] \times \left(\frac{5.8}{2}\right)^{2.5} \times 2.23 =$$

$$1.99 \times 14.3 \times 2.23 = 64 \text{ net hp.}$$

Actually, this mill takes 67 net horsepower.

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[8] A. M. Gow, A. B. Campbell and W. H. Coghill: Reference of footnote 1, 24.

Example 2.—The same mill was run with 37 per cent mill volume of balls and at 79 per cent speed. The basic curve gives P as 1.96, and substituting in the formula gives:

$$1.99 \times 14.3 \times 1.96 = 56 \text{ net hp.}$$

Actually, the mill was taking 54 net horsepower.

Example 3.—A 6½ by 12-ft. rod mill was converted to a ball mill, loaded to 45 per cent mill volume with balls, and run at 67 per cent speed. The feed rate was 42 tons per hour, and the mill, being originally a rod mill, had a large discharge opening. Actual inside dimensions were 6.2 by 12.2 feet.

The base curve gives P as 1.74. Although the large discharge trunnion calls for an increase of about 8 per cent, according to Fig. 4, the feed rate is relatively large, and the length of the mill tends to counteract the draining effect of the large discharge. Consequently, the above value is used without correction. Substituting in the equation gives:

$$\begin{aligned}\left[\left(\frac{12.2}{2} - 1\right) \times 0.85 + 1\right] &\times \left(\frac{6.2}{2}\right)^{2.5} \times 1.74 = \\ 5.3 \qquad\qquad &\times\ 16.9 \qquad \times 1.74 = 156 \text{ net hp.}\end{aligned}$$

Actually, the mill used 205 gross hp., which, assuming a 25 per cent dead load and motor loss, would give 155 net horsepower.

Example 4.—What size motor should be used if the above mill is to be run at its maximum capacity? The basic curve gives this maximum value as about 2.4 net hp. Substituting as above:

$$5.3 \times 16.9 \times 2.4 = 215 \text{ net hp.}$$

Again, assuming a 25 per cent dead load and motor loss, a motor of at least 290 continuous horsepower should be used. This estimate should not be construed as advocating such high-speed operation or as overlooking "starting load."

Example 5.—A 6 by 4-ft. mill is loaded to 50 per cent mill volume of balls and run at 60 per cent speed. The gross power consumption is 61 hp. How much total power will be used by a 6½ by 12 ft. mill loaded with 40 per cent mill-volume balls and run at 80 per cent speed, other conditions being analogous? The inside dimensions are as quoted in the former examples.

By referring to the basic curves the relative net powers under the two sets of operating conditions are found to be 1.6 for the smaller mill and 2.1 for the larger mill. The net power for the larger mill will be greater than that of the smaller mill by the increase in length, by the ratio in diameter raised to the 2.5 power, and by the increase due to operating conditions. Again, dead load and motor loss will be assumed as 25 per cent of the gross in both cases, and the gross power may be calculated directly, thus:

$$61 \times \frac{12.2}{4.2} \times \left(\frac{6.2}{5.8}\right)^{2.5} \times \frac{2.1}{1.6} =$$

$$61 \times 2.9 \times 1.2 \times 1.3 = 275 \text{ gross hp.}$$

Example 6.—A 4 by 10-ft. ball mill is loaded with 9 tons of balls and run at 29.4 r.p.m. The discharge opening is 10¼ in. but, in order to retain the balls, an extending perforated housing was mounted on the discharge, and this is over half full of balls when the mill is running. The inside dimensions of the mill are 3.6 by 10.2 feet.

The ball volume is 58 per cent and the speed 73 per cent of the critical. From Fig. 3, value P would be 2.10 hp., because ball-volume values above 55 per cent use the 55 per cent curve. However, the mill is long and has a small, constricted discharge opening, therefore the value must be reduced because of swelling of the charge. Use 2.0 for the estimate. Substituting in the formula gives:

$$\left[\left(\frac{12.2}{2} - 1\right)0.85 + 1\right]\left(\frac{3.6}{2}\right)^{2.5} \times 2.0 =$$

$$4.34 \times 4.35 \times 2.0 = 38 \text{ net hp.}$$

A recording wattmeter showed the gross horsepower to be 48.5. The net, then, is actually about 36 horsepower.

Additional comparisons between the observed and estimated power for several operating mills are given in Table 1.

Power to Start a Mill

Mention has been made of the starting load on a ball-mill motor. Except where the sand has packed within an idle mill, there is no inherent peak load upon starting a ball mill. Sudden starting will cause a peak because of the quick acceleration; but it is possible, when facilities are adequate, to bring a mill slowly up to speed, to reach maximum operating speed without exceeding maximum operating power.

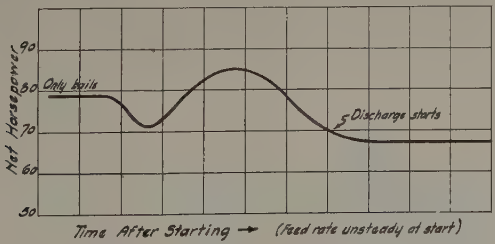

Fig. 10.—Power of a 6 by 4-ft. ball mill while it was being filled with pulp; 50 per cent ball volume, 9-in. discharge opening.

Some additional power data were gathered upon starting a 6 by 4-ft. mill which had a 9-in. discharge opening and was loaded to 50 per cent mill volume with balls. Power readings were taken while the running mill, charged with balls alone, was gradually filled with ore and water to its normal overflowing capacity. During this time of filling, the power rose to a maximum and then dropped to the constant value of continuous operation. The curve of power plotted against time, Fig. 10, shows this rise and fall. If the discharge opening had been large enough to reduce the mill's pulp content to the quantity that required maximum power, the operating net power would have been about 25 per cent greater than what was ultimately consumed, and the power curve would run horizontally from its present peak.

TABLE 1.—*Comparison of Observed and Estimated Power for Various Mills*

Size of Mill, Ft.		Ball Volume, Per Cent	Speed, Per Cent	Net Horsepower[a]	
Nominal	Actual			Observed	By Formula
4 × 10	3.6 × 10.25	58	66	29	33
		58	73	36	38
6 × 4	5.8 × 4.2	50	80	67	64
		50	72	60	56
		50	69	56	54
		50	63	49	48
		50	60	46	45
		37	79	54	56
		31	79	46	50
		28	79	41	46
		25	79	38	40
6 5 × 12	6.1 × 12.1	41	52	110	111
		41	60	129	133
		41	67	148	152
		45	60	138	136
		45	67	154	156
		50	60	134	142
		50	67	160	162
		55	60	140	150
		55	67	160	170
		59	60	136	150
		59	67	160	170
8 × 6	7.7 × 6.0	51	84	190	182

[a] Net observed horsepower was obtained by taking 75 per cent of the gross power reading, when dead-load data were not available.

Operating Variables in Grinding

Grinding capacity and grinding efficiency are the ultimate values which interest the operator and which must be increased, either separately or together as the case may demand, so that ball milling may be improved. Capacity is expressed in surface tons per hour and efficiency in surface tons per net (or gross) horsepower-hour. In each case, only useful grinding is evaluated; the calculations are carried down to a limiting size beyond which additional grinding is unwarranted. The limiting or "useful" size will vary with different plant requirements, but in this investigation two sets of calculations have been used, those through 65-mesh and those through 200-mesh. Since the method of making these calculations has been discussed elsewhere,[9] no further explanation is necessary here.

All the operating variables affect the capacity and efficiency of grinding, but for a thorough understanding of their effects they must be divided into two groups, set variables and induced variables. The set variables are those that are not changed by other operating conditions and include the size and design of the mill, the size of the discharge opening, the type of lifters, the speed of rotation, the amount and size of the ball charge and the type of feed. Also, the feed rate, the sizing analysis of the feed, and the pulp density generally may be considered in this class.

On the other hand, the induced variables are such factors as the amount of ore in the mill, the location of the center of mass of the ball-and-pulp charge, the amount of slippage, the power required and, in closed-circuit grinding, the type of the circulating load.

A variation in one of the set variables might tend to produce a certain change in the capacity or efficiency of a mill, but also it might result in changes in an induced variable, which would either magnify or diminish the primary tendency. Were it not for these induced variables, the investigation of ball milling would be a simple matter; the different set variables could be studied to find the conditions of best speed, of best mill volume of balls, of best circulating load, and so on. However, both plant practice and laboratory work have shown that such a test procedure is not comprehensive.

A series of grinding tests at various speeds, when the feed rate is constant, gives products that are all different in fineness, making the evaluation of grinding difficult. To avoid this and to make the amount of ore to be ground commensurate with the available ball action a standardization of *tons per horsepower-hour* has been found desirable in the laboratory. In the field, too, this unit proves convenient in analyzing stage grinding and in determining grindability.

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[9] W. H. Coghill: Evaluating Grinding Efficiency by Graphical Methods. *Eng. & Min. Jnl.* (1928) **126**, 934–938.

A. M. Gow, A. B. Campbell and W. H. Coghill: Reference of footnote 1, 18.

"Tons per horsepower-hour" is distinctly for the ore dressing engineer. Similarly, its counterpart, "horsepower-hours per ton," is for the accountant. In the first, "tons" applies to the composite or actual feed to the mill, while in the second, which is designed for the accountant who is interested in over-all plant results, the value is expressed in tons of new feed. Horsepower-hours per ton of new feed give little assistance in a purely metallurgical analysis and consequently are not used in this paper.

For the laboratory tests the values 0.6 and 0.2 ton per horsepower-hour have been adopted as the feed rates for dolomite and flint that would give satisfactory products for experimentation. Moreover, these values are typical of good plant practice.

Amount of Pulp in Mill

In batch grinding there can be found a certain charge of ore that results in the maximum capacity or efficiency, but in continuous grinding the amount of ore in the mill is an induced variable that depends on several set operating conditions. It must be given consideration, as has been stated before. In the laboratory the amount of pulp in an operating mill has been ascertained by suddenly stopping the mill, catching the surge of effluent, and then weighing the entire mill, contents, and spill.

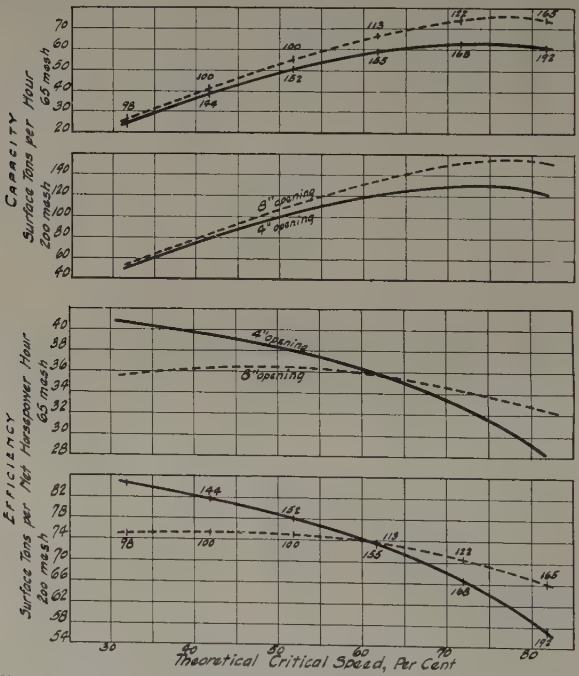

FIG. 11.—CAPACITY AND EFFICIENCY VALUES AND AMOUNTS OF ORE IN MILL FOR GRINDING TESTS AT VARIOUS SPEEDS WITH 4 AND 8-IN. DISCHARGE OPENINGS ON THE 2 BY 2-FT. MILL; 45 PER CENT BALL VOLUME.

A series of grinding tests was made with dolomite, in which all set variables were constant except the speed and feed rate. The tonnage per horsepower-hour was constant. The 2 by 2-ft. mill was equipped with a 4-in. discharge opening. The purpose was to study grinding capacity and efficiency at various speeds. The results are shown in the first half of Table 2 and by the full lines in Fig. 11.

From these data the conclusions may be drawn that a high speed, not in excess of 72 per cent of the critical, favored capacity, but a low speed favored efficiency. These, in general, are the conclusions drawn from former investigations and are true for a mill run under these conditions. However, the high speed, in itself, should not be blamed for the low efficiency that accompanied it, because other tests have shown that high-speed grinding can be just as efficient.

A second series of grinding tests was run exactly like the first, except that an 8-in. discharge opening was used. The results of this series with the larger discharge opening are given in the second half of Table 2 and by the dotted lines in Fig. 11. The numbers on the curves represent the pounds of ore in the mill.

TABLE 2.—*Grinding Capacities and Efficiencies at Various Speeds and Various Amounts of Pulp in Mill with Four-inch and Eight-inch Discharge Openings*

	Four-inch Discharge Opening					Eight-inch Discharge Opening					
Speed, per cent critical	42	52	62	72	82	32	42	52	62	72	82
Capacity:											
Surface tons per hour (65-mesh)	39.9	48.7	59.2	63.8	61.5	26.6	42.1	54.4	65.9	74.3	74.1
Surface tons per hour (200-mesh)	83.1	100.3	120.3	130.4	125.0	56.1	87.4	112.7	137.1	154.2	153.0
Efficiency:											
Surface tons per net hp-hr (65-mesh)	39.5	36.6	36.0	32.9	28.2	35.7	36.3	36.3	35.4	34.3	32.3
Surface tons per net hp-hr. (200-mesh)	82.2	75.4	73.3	67.2	57.3	75.3	75.3	75.1	73.7	71.0	66.0
Pounds of ore in the mill	144	152	155	168	192	98	100	100	113	122	165

The conclusions to be drawn from this second series are that high speed, not exceeding 72 per cent of the critical, favors capacity, as before, but that with proper conditions of operation high speeds may give as good efficiency values as low speeds. In this case the efficiency values are nearly constant. A horizontal efficiency curve would indicate that the amount of grinding was directly proportional to the power expended, and these tests suggest that such a condition can be made to exist in commercial operations.

From the first glance at Fig. 11 one might reason that if the 8-in. opening gives better high-speed efficiency than the 4-in., possibly a still larger opening would give an even better value. However, the intersection of the two curves would indicate that the best amount of ore lies roughly between 113 and 155 lb. and that a discharge opening that was so large as to retain only a small amount of ore would be inefficient also. The latter was proved by using an 18-in. opening. The efficiency values were lower than those for the 4-in. discharge.

The practical significance to the operator is that an overflow-type mill with a small discharge opening may be inefficient because it holds too much ore within the mill, and a low pulp-level mill may be equally inefficient if it discharges so readily that too little ore is retained in the mill. Unfortunately, one correct size of opening for an overflow-type mill or one correct pumping rate for a low pulp-level mill cannot be predetermined for general application, because, as stated previously, the induced variables depend on many set factors of operation.

Speed, Impact and Attrition

Certain variations due to speed have been discussed. Provided other operating conditions are favorable, a high speed, when it results in a high power, may be expected to give both high capacity and efficiency.

The idea that the product of high-speed grinding differs from that of low-speed grinding has been proved erroneous, as is shown by sizing analyses of products from similar laboratory tests at low speed and at high speed. The feed was minus ⅛-in. dolomite, the feed rate was 0.6 ton per net horsepower-hour, and the discharge opening was 8 in. The

TABLE 3.—*Sizing Analyses, Showing Similarity of Products of Low-speed and High-speed Grinding, When Tonnage per Horsepower-hour Was the Same*

Size Mesh	Weight, Per Cent		
	Feed	Discharge	
		32 Per Cent Speed	72 Per Cent Speed
8	0.9	0.3	0.2
10	14.4	3.7	2.5
14	35.5	11.1	9.8
20	28.7	15.6	16.2
28	10.7	12.9	14.1
35	5.2	11.7	13.0
48	1.9	7.7	8.2
65	0.9	6.4	6.5
100	0.4	5.0	4.9
150	0.2	4.4	4.2
200	0.2	3.6	3.4
−200	1.0	17.6	17.0
	100.0	100.0	100.0

sizing analyses are shown in Table 3. The product resulting from the "attrition" grinding at 32 per cent speed is almost identical with that from the "impact" grinding at 72 per cent speed.

Rationing Ball Size to Grain Size and Hardness

The old idea that hard ore should be broken by impact grinding led to the impression that hard ore should be broken by large balls. If an operator whose mill was set for grinding dolomite were to plan on grinding flint in the same mill he might be inclined to increase the ball size. This would be rationing the ball size to the grindability of the ore. Rationing the ball size to the ore size is common practice.

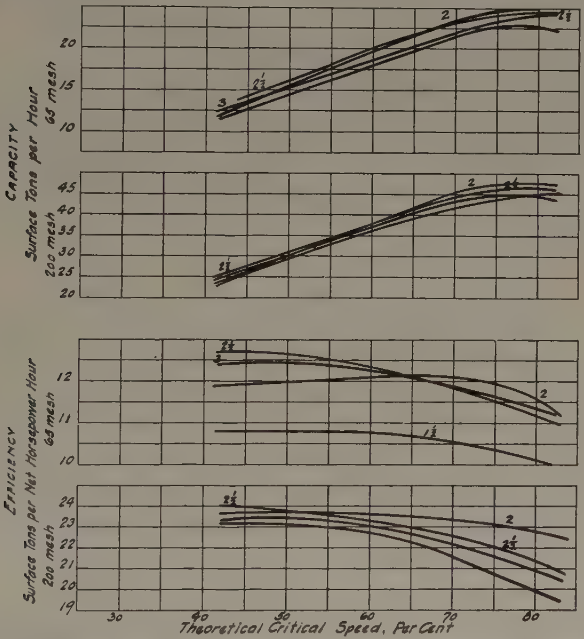

FIG. 12.—CAPACITY AND EFFICIENCY VALUES OF GRINDING TESTS AT VARIOUS SPEEDS AS MINUS 6-MESH FLINT WAS GROUND WITH BALLS OF VARIOUS SIZES; 45 PER CENT BALL VOLUME.

No attempt will be made here to state the maximum size of ball that should be used on every size and kind of feed, but a few laboratory and field tests deserve mention because they show that ball size may be smaller than is generally expected and that the hardness does not make much difference, except in the grinding rate.

In the laboratory, two extensive series of grinding tests were made; in one, hard, Tri-State flint was used, and in the other, soft Southeast Missouri dolomite was the feed. Both feeds were of the same grain size, minus 6 mesh with 50 to 55 per cent on 14 mesh and 2 per cent through 65 mesh. Ball charges of four sizes were employed: 1½-in. maximum, 2-in. maximum, 2½-in. maximum, and 3-in. maximum. Each ball charge was graded down to ¾-in. minimum size ball, and each was used in grinding tests throughout the speed range of 32 (or 42) per cent to 82 per cent of the critical speed.

The feed rate for the flint was 0.2 ton per net horsepower-hour, and for the dolomite, which has nearly three times the grindability, the rate was 0.6 ton per net horsepower-hour. Pulp density was maintained at 60 per cent solids by weight for the flint and 70 per cent solids for the dolomite. The mill was the 2 by 2-ft. ball mill with a 4-in. discharge opening. The data are portrayed in the curves in Figs. 12 and 13.

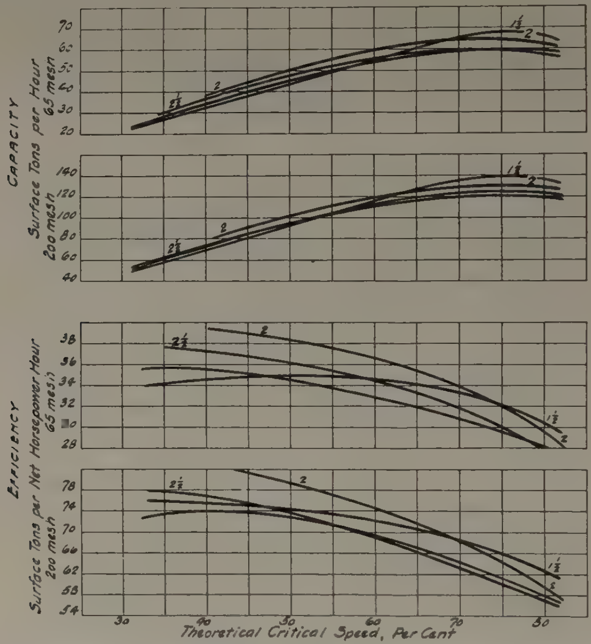

FIG. 13.—CAPACITY AND EFFICIENCY VALUES OF GRINDING TESTS AT VARIOUS SPEEDS AS MINUS 6-MESH DOLOMITE WAS GROUND WITH BALLS OF VARIOUS SIZES; 45 PER CENT BALL VOLUME.

The sizing analyses of the products made by the 2½-in. balls have been selected for illustration and are shown in the upper part of Table 4, and in the lower part are shown accompanying data. The similarity of the grain size at the different speeds is brought out more fully than in Table 3. In fact, in spite of the old impact and attrition theories, the grain sizes are so uniform that surface calculations are scarcely necessary to diagnose the results. If the early experimenters had sensed the importance of standardizing their tests in terms of tons per horsepower-hour the incentive to argue about Rittinger-Kick would have been less.

In the grinding of flint the highest capacity and the highest efficiency values were obtained by using 2½ to 3-in. maximum size balls at slow speed and 2 to 2½-in. maximum size balls at high speed. In the grinding of the dolomite slightly smaller balls sufficed; the 2 to 2½-in. maximum balls at slow speeds and the 1½ to 2-in. maximum balls at high speed gave the best results. In other words, hard ore and slow speed require slightly larger balls than soft ore and high speed. As far as practical ball milling is concerned, however, the variation in ball size to compensate for hardness or softness of ore and for slow or high speed is so small that the operator might say with justification that neither the hardness of the ore nor the speed of the mill makes any appreciable difference in setting the correct sizes of ball, and balls of 2 to 2½-in. maximum diameter are very effective in grinding minus 6-mesh ore.

Table 4.—*Sizing Analyses of Feed and Discharge in Open-circuit Grinding with 2½-inch Balls and at Various Speeds When Flint and Dolomite Were Ground at Rate of* 0.20 *and* 0.60 *Ton per Horsepower-hour, Respectively*

Size Mesh	Flint						Dolomite						
	New Feed Weight, Per Cent	Critical Speed, Per Cent					New Feed Weight, Per Cent	Critical Speed, Per Cent					
		42	52	62	72	82		32	42	52	62	72	82
8	1.7	0.2	0.2	0.2	0.2	0.2	0.9	0.2	0.2	0.1	0.2	0.2	0.1
10	23.5	2.8	2.8	2.9	3.4	3.2	14.4	2.5	2.1	2.1	2.2	2.6	3.6
14	29.8	6.2	6.6	6.8	7.8	8.0	35.5	9.3	8.7	9.6	10.3	11.7	14.6
20	21.7	10.5	10.9	12.1	12.9	14.0	28.7	15.1	14.7	16.5	17.6	19.0	21.4
28	10.5	12.1	12.5	13.2	14.0	14.4	10.7	13.5	13.5	14.6	14.8	15.0	15.0
35	6.2	14.6	15.0	14.8	15.0	14.9	5.2	12.9	13.1	13.4	13.5	12.8	11.9
48	2.8	11.2	11.2	10.8	10.6	10.2	1.9	8.4	8.7	8.3	8.1	7.6	6.5
65	1.7	10.0	9.8	9.1	8.9	8.5	0.9	6.9	7.0	6.5	6.4	5.8	4.9
100	1.0	7.8	7.1	7.1	6.7	6.5	0.4	5.3	5.4	4.9	4.6	4.3	3.5
150	0.5	5.7	5.5	5.2	4.8	4.7	0.2	4.7	4.6	4.1	3.9	3.6	3.0
200	0.2	3.9	3.7	3.5	3.2	3.0	0.2	3.6	3.6	3.3	3.1	2.9	2.4
−200	0.4	15.0	14.7	14.3	12.5	12.4	1.0	17.6	18.4	16.6	15.3	14.5	13.1
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100 0
Surface tons, hp-hr. (65 mesh)		12.7	12.6	12.2	11.6	11.1		37.6	37.8	34.1	32.6	31.1	27.0
Surface tons hp-hr. (200 mesh)		24.0	23.8	23.1	21.3	21.1		77.6	78.6	70.2	66.2	57.9	56.0
Tons per hour		0.201	0.260	0.336	0.408	0.441		0.420	0.614	0.795	1.000	1.210	1.330
Horsepower		1.05	1.33	1.71	2.02	2.21		0.68	1.01	1.33	1.65	1.95	2.12
Tons per horse-power hour		0.19	0.20	0.20	0 20	0.20		0.62	0.61	0.60	0.61	0.62	0.63

After completion of these tests 2-in. balls were tried for similar work in the field and were found satisfactory. In one plant, when coarse grains 3-mesh to ½ in. in size are accidently mixed with the feed, this material is temporarily shunted to a mill containing larger grinding media because it has been found that 2-in. balls cannot grind such coarse material effectively. In another plant (See Table 10) the circulating load of 6-mesh grains is larger than the circulating load of 8-mesh grains. The amount and size of the circulating load will be discussed under an appropriate heading.

Improper rationing of the ball size to a particular grain size is shown by two laboratory closed-circuit tests, in which ¾-in. balls were compared with a mixture of 3-in. and 2½-in. balls. The data in Table 5 show that the small balls failed to grind much of the plus 10-mesh material and overground the fines; but in spite of this they were better than the large balls, which crowded the nearly finished material and thereby increased the circulating load without increasing the total amount finished. The two ball charges represent extremes in size.

TABLE 5.—*Sizing Analyses and Tonnage Data, Showing Effects of Extremes in Ball Sizes*

Size Mesh	Weight, Per Cent				
	New Feed	Circulating Load		Finished Product	
		Small Balls	Large Balls	Small Balls	Large Balls
6	6.2	3.0	0.1		
8	10.6	12.4	0.6		
10	8.3	11.4	0.9		
14	6.7	8.6	1.1		
20	6.4	6.5	2.4		
28	6.8	5.9	4.6		
35	17 6	10.5	18.8		
48	20.5	16.2	38.8	1.5	4.2
65	12.5	15.5	23.6	6.5	19.9
100	3.8	6.7	6.2	14.0	19.9
150		2.2	1.7	15.5	14.6
200	0.6	0.6	0.6	11.1	8.9
−200		0.5	0.6	51.4	32.5
Ton per hour		0.120	0.423	0.156	0.147

When once a feed has been prepared as the best product for the fine-grinding section it becomes one of the few inflexible elements of the grinding circuit. The ball charge should be selected to impart a well-rationed amount of its grinding to each size, then the nearly finished material will neither be overground nor allowed to build up excessively. This gradation through many sizes permits good classification because over-accumulation of the nearly finished size makes washing out the fines difficult. A circulating load should be so similar to the new feed in grain-size distribution that the balls that grind the new feed efficiently can work with nearly equal efficiency on the circulating load. This will be discussed further when closed circuits are considered in more detail.

Amount of Ball Charge

The amount of grinding media may be too small or too large for good grinding. Small charges slip in the mill and necessitate higher shell speeds for a given amount of ball action than if there were no slipping, resulting in a proportionally higher dead load and a consequent low over-all efficiency. In commercial operation large charges of 55 to 60 per cent of the mill volume have been found to give lower capacities and efficiencies (see Fig. 16) than loads of 45 to 50 per cent. Between the ranges of extremely large and extremely small charges the capacity and power consumption vary with the ball charge. As the efficiency depends largely on induced variables it will depend on other operating conditions. A run made without balls and while feeding ore and water used about one-third the power required for normal grinding. With flint practically no grinding was accomplished by the tumbling. Obviously, a small charge of balls could not be expected to do enough grinding to make up for both the power of ball action and that for tumbling the ore.

Size and Design of Mill

The advantage of a large mill over a small mill lies in its ability to use more power and do more grinding. In the correlation of the performance of small and large mills in the laboratory and field the advantage lies with the small mills, but the difference is slight.

For a given grinding capacity a mill might well be designed so that the length is proportional to the relative hardness of the ore. This relationship will receive further discussion when heterogeneous ores are considered (Table 8).

The liners in a mill are used to take up the wear inside the shell. Since this wear is due primarily to slipping, liners are generally designed to reduce sliding of the charge. Sometimes lifter bars are used; but in any case, the joints between the sections and the other irregularities in the lining tend to reduce slipping of the charge. It would be almost impossible to build a smooth lining in a large mill. The degree to which the liners or lifters stop slipping is allied closely with the amount of the ball charge. Large ball volumes are not as prone to slipping as small ones, and with them the effect of lifters has less importance.

A certain amount of grinding is done between the ball charge and the liner surface; the rest is done between the balls. The relative amount of ball surface and shell surface may be important in the design of the mill. The use of rubber linings practically nullifies the shell area as a grinding surface, but in spite of this rubber has been reported by other investigators as satisfactory.

The normal mixture of balls, sized from 2½ to ¾ in., has a surface area of about 26 sq. ft. per cubic foot of balls. This value was ascertained by counting and measuring the balls of the charge used in the laboratory mill. In the 2 by 2-ft. mill one-half of the total shell area—the part in service at any instant—represents about 8 per cent of the ball area with 50 per cent ball volume. However, with a large mill the ratio is smaller, and with a 6 by 12-ft. mill under the same conditions it is less than 2 per cent. If none of the lining of a large mill were available as grinding area the reduction in grinding would be insignificant. The inference is, then, that if rubber lining increased the amount of grinding the increase was due to reduced slipping, which resulted in more ball action.

Pulp Density

No simple rule can be stated regarding proper pulp density. The literature invariably states that the pulp in a pebble mill should be thin, lest the pebbles tend to float, but beyond this evident fact there is little agreement. Pulp density is regulated at times by the noisiness of the mill or the temperature of the discharge.

The idea is generally held and quoted that "the pulp should be thick enough to cling to the balls but not so thick as to cushion the blows." This statement infers that the water will wash the balls clean if there is an opportunity. The reality is that the intense showering of wet sand in all directions in the otherwise unoccupied spaces of a ball mill relieves the "clinging" theory from having to account for grinding. Also, observers of the pool at the toe of the heap of balls have held that a thin pulp would allow slime to flow directly to the discharge and result in self-classifying action by the ball mill. Both conditions have been investigated by using a cup on the end of a long handle to sample the pulp inside a running mill. The mixing was found to be so thorough that even skimmings from the pool were of the same grain size as the shower of sand that fell just inside and above the outermost layer of balls. No segregation of ore from water could be found within the mill, and the pulp density inside was the same as that of the effluent.

In the majority of the tests reported herein the pulp density was constant in each series. Relatively few tests were made to study its effect on grinding, but many tests were made to ascertain how pulp density affected power. The change in grinding or power attributable to variation in pulp density is small, but pulp density has a strong influence on the induced variables. The amount of ore in the mill depends to a considerable extent on the pulp density.

If the pulp is dilute the mill contains less ore, and for a given rate of feed the ore passes through the mill faster. This increased rate of travel tends toward better stage grinding. But there is a compensating factor; the ball action is concentrated on a smaller amount of ore, producing a tendency to overgrind. The ultimate results have to be found by trial. When two laboratory tests were run under similar conditions, one with a pulp density of 45 per cent of solids and the other at 70 per cent, there was only a slight difference in the results. The thick pulp used 2 per cent more power and gave 4 per cent more surface tons per hour (65-mesh).

If a mill is run at moderate speed with a dilute pulp and the pulp is gradually thickened the power will rise because the thicker pulp reduces slipping and there is consequently increased ball action. This phase of pulp density was discussed under "slipping." Thickening the pulp causes the ore to build up in the mill, and if the size of the discharge trunnion is small the ore will build up so much that the large amount shifts the center of mass of the composite charge toward the center of the mill to reduce the power. In any event, the pulp finally will be thickened to the point where the mill will become choked and ball action will cease.

Another phase of pulp density pertains to the density of the solids themselves. For equal volumes of pulp, those of heavy minerals use more power than those of flint or dolomite. When the amount of ore is only sufficient to fill the interstices of the ball charge at rest (38 per cent) the proportional part of the net power chargeable to a flint pulp is about 13 per cent, and for a pyritic pulp it is about 21 per cent.[10] This percentage of the power charged to imparting action to ore mounts rapidly as the mill fills with more than enough ore to occupy the interstices.

Feed Rate

The effect of the feed rate on the amount of ore within the mill has been discussed, but another aspect of the subject must be mentioned. Just as a feed rate that is too small results in overgrinding and correspondingly low values in useful capacity and efficiency, so a large feed rate tends to increase the amount of useful grinding. The feed rate should be large enough so that some coarse material still remains by the time the ore reaches the last section of the mill. Otherwise, the balls will waste their energy in overgrinding pulp that was sufficiently ground in the earlier part of its passage through the mill.

A pair of tests were run in the laboratory which showed the increase in useful grinding as the feed rate was increased. In this instance "useful" grinding was figured at three sizes, 200 mesh, 65 mesh and 28 mesh. The data, together with a column showing the percentage of increase, are presented in Table 6.

The feed rate was increased 81 per cent, but the increase in useful grinding was only from 11 to 16 per cent, depending on the limit of the sizes credited to useful grinding. The increase is small compared with the increase in feed rate. If the mill had been credited for the very finest sizes no advantage from increased feed rate would have accrued.

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[10] For convenience the density of the flint and the pyritic pulps are taken as 2.0 and 3.4, respectively.

The slight variation in power agrees with what was said in discussing power (Fig. 5), previously; it was reduced slightly by the larger feed rate

Circulating Load and Classification

Circulating load is sometimes regarded as the panacea for the ills of ball milling, and attempts have been made to solve the fine-grinding problem by ascertaining the correct amount of material that should constitute the circulating load.

TABLE 6.—*Increase of Useful Grinding with Increase in Feed Rate*

	Test A	Test B	Increase, Per Cent
Feed rate, ton per hour	0.189	0.342	81
Net horsepower	1.01	0.99	
Ton per net horsepower-hour	0.187	0.346	
Capacity:			
Surface tons per hour (200 mesh)	23.4	25.8	11
Surface tons per hour (65 mesh)	12.5	14.2	13
Ton through 28-mesh per hour	0.106	0.121	14
Efficiency:			
Surface tons per net horsepower-hour (200 mesh)	23.2	26.0	12
Surface tons per net horsepower-hour (65 mesh)	12.4	14.3	15
Ton through 28 mesh per net horsepower-hour	0.105	0.122	16

In a hurried passage through a ball mill some of the feed becomes sufficiently ground while the rest is still oversize. With a slow feed rate grinding could proceed until all had reached the finishing size, but this would result in overgrinding, which is wasteful and often harmful. Closed-circuit grinding must be a compromise between an attempt to grind for capacity's sake and an attempt not to grind too much lest the result be overgrinding.

The grains ground to the required size should be removed immediately and replaced by new feed. Theoretically, the separation of finished from unfinished should be coincidental with the grinding. Practically, this is impossible. The grinding and subsequent classification must be periodic, and the duration of each grinding period determines the amount finished in each pass and the amount of circulating load. Thus, when one-half of the feed is ground out in one pass the circulating load is 100 per cent; when one-third is ground out the circulating load is 200 per cent; when one-fourth is ground out the circulating load is 300 per cent; and so forth.

The circulating load may be considered one of the operating variables, inasmuch as it changes the character and tonnage of the composite feed to the mill. When certain variables, such as ball size and mill design, are made to conform to the feed they should be set with respect to the composite or actual feed, not merely to the new feed if the circuit is closed; or, as has been pointed out previously, the grain size of the circulating load should be similar to that of the feed so as to be rationed to the ball charge.

In commercial practice the classifier is often the bottle neck in the circuit, and the large tonnages that might be indicated for good grinding cannot be handled by the accessory apparatus. In other cases the efficiency of the classification is so poor under heavy tonnage that stage grinding suffers in spite of a large circulating load. In this connection the claim has been made in the literature that classification can be too thorough and that allowing a certain amount of finished material in the feed is beneficial to the grinding despite the resulting overgrinding. This idea is contradicted, and the importance of a quick, thorough removal of finished material is emphasized by a pair of laboratory tests. In one, coarse dolomite was ground under standard conditions; in the other, the grind was repeated with fine silica sand added to the feed. The mill discharge from the second run was leached, and the sizing analysis of the dolomite was found by difference. The two dolomite products, one ground by itself and the other fed at the same rate with finished material present, are shown in Table 7. The grinding of dolomite was retarded by the presence of the sand of finished size. In those instances in commercial operation where it is reported that the presence of slimes in the feed was an aid to grinding, no mention is made of the relative power expended or of the degree of inefficiency.

TABLE 7.—*Comparison of Ground Products When Dolomite Was Ground Alone, and in the Presence of Sand of Finished Size*

Size Mesh	Dolomite Ground Alone, Per Cent	Dolomite in Presence of Finished Sand, Per Cent
6	0.2	0.2
8	1.4	3.0
10	2.2	3.8
14	3.9	5.6
20	5.1	6.8
28	5.8	8.1
35	7.8	11.1
48	7.4	11.6
65	8.0	10.0
100	7.5	8.6
150	7.4	6 3
200	7.0	5.0
−200	36.3	19.9
	100.0	100.0

The classifying circuit cannot be divorced from the closed-circuit ball mill. The interdependence of the two is so great that the full benefits of any improvement in one cannot be fully realized in the face of an inefficiency of the other. The authors do not know any means of good desliming without an abundance of wash water. When the present drag deslimers were introduced it was expected that wash water would be added to the sand, but this plan only aggravated the conditions by washing the sand back into the tank and increasing the amount of sand from which the slime had to be removed. So, wash water fails in the drag deslimer, and as a result the separation is poor; but if condemnation has to be evoked, it should not fall too heavily on the drag but upon the operator who expects too much of it. The drag should be considered only the first unit in the classifying system and should be supplemented by a more efficient system of separating finished from unfinished grains. This procedure has been perfected to a degree that would surprise some operators. It includes what in many of the mining districts is today a lost art; namely, tabling and hydraulic classification.

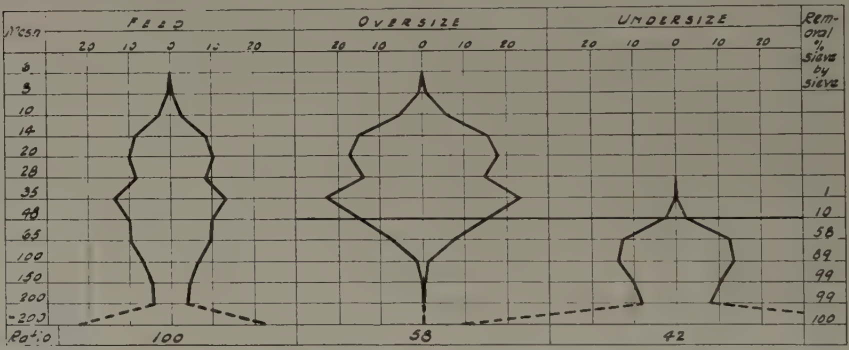

FIG. 14.—SIZING DIAGRAMS OF PRODUCTS OF A 48-MESH SEPARATION MADE WITH A DRAG DESLIMER, HYDRAULIC CLASSIFIER AND TABLES.

The authors have had a hand in the development of this system from its beginning to its perfection for 25,000 tons daily. Although it was developed for betterment of tabling and preparation of the ball-mill feed was incidental, it would work just as well if the classification were made the primary object.

Tables and multiple spigot hydraulic classifiers for desliming would have their greatest success in closed circuit with a ball mill when the finishing is anywhere from 28 to 150 mesh and when the ore contains a heavy mineral that can be recovered on the tables, either as concentrates or as a feed for treatment in an isolated circuit. The thoroughness of this kind of sizing operation was demonstrated in the plant by splitting the products by hand into oversize and undersize at approximately 48 mesh to simulate regular table cuts. Sizing diagrams of these hand-made products are shown in Fig. 14. The oversize is a composite of the coarse spigot products and the coarse table cuts of the medium spigots. The undersize is the composite of the fine table cuts of the medium spigots, the fine spigot products, and the overflows of the classifier and the drag. The efficiency of the separation is shown by the column "per cent removal." The product was a dolomitic lead ore containing locked middling grains, but the heavy sulfides were not included. The classifier was not set primarily for a separation at this size. The amount handled by the 10-spigot classifier and tables was 700 tons per day.

The scheme has not been used in all-flotation work, but it would be equally amenable. The present practice of making flotation feed by the overflow of a classifier allows no accurate control of grain size. Heavy grains must be excessively fine to flow with the light ones; and the operator must accept the product of the fine-grinding section, once the limiting size has been established. In the proposed system of closing the circuit the story is entirely different. Grains of any size and specific gravity may be removed from the circuit by cutting them from the table on which they appear. Control of almost theoretical precision is made possible. The objection that arises from a large table room need not be serious, for only spigot products that carry the desired mineral need be tabled; the rest may be bypassed from the hydraulic classifier to the ball mill or the thickener. It is possible that if this were done only three or four tables would be needed for 15 tons of new feed per hour.

With a method of thoroughly removing the finished material from the circuit the amount of circulating load required for good stage grinding may be approached more logically. The authors believe that if, in one pass, one-half of the coarsest grains were reduced to finished size (100 per cent circulating load) and one-fourth of the nearly finished grains were ground out (300 per cent circulating load) the stage grinding would be satisfactory. With efficient classification and a ball charge that would ration the grinding in the manner just stated, this would mean a composite circulating load of about 200 per cent. In reality, even though the actual load would be small, this would give better stage grinding than is generally found in practice, where large circulating loads carry finished material around and around.

Grindability of Heterogeneous Ores

Much of what has been said thus far applies only to the grinding of homogeneous ores, but generally the valuable mineral in an ore is of a different hardness or grindability from the gangue. Heterogeneity of the feed throws another aspect on ball milling.

A ball mill, unlike rolls and most other crushing machinery, is a differential grinder. The capacity for grinding is inversely proportional to the grindability of the material ground. If a hard mineral and a soft mineral are mixed and fed to a set of rolls they become about equally ground, but if the mixture is fed to a ball mill the softer mineral suffers much more reduction than does the harder one. Table 8 shows the sizing analyses of flint and dolomite of equal size, mixed in equal proportions and then ground in both rolls and a ball mill. The sizing analyses were made after the minerals were separated with a heavy liquid.

As has been pointed out before, dolomite has about three times the grindability of flint, as expressed in tons per net horsepower-hour that will give the same products. Expressed a little differently, a flint mill 4 ft. long would seem 12 ft. long to dolomite, and a mill 12 ft. long would grind flint as a 4-ft. mill would grind dolomite. When a hard and a soft mineral are ground together in a ball mill, no matter how great the degree of stage grinding, the softer mineral is the more overground. Of course, if the circuit is closed with a classifier and the soft mineral is the heavier the soft mineral has to submit to being overground to overflow the classifier, anyway, but this does not justify the practice of overgrinding.

Table 8.—*Comparative Grinding Rates of Mixed Chert and Dolomite in Rolls and in a Ball Mill, as Shown by Sizing Analyses*

Size Mesh	Mixed Feed to Rolls, Per Cent	Distribution in Roll Discharge		Mixed Feed to Ball Mill, Per Cent	Distribution in Ball-mill Discharge	
		Chert, Per Cent	Dolomite, Per Cent		Chert, Per Cent	Dolomite, Per Cent
0.371 in.	11.9	0.8	0.7			
3 mesh	46.8	6.6	7.2			
4	41.3	38.4	36.8			
6		24.2	22.2	43.2	26.5	11.1
8		10.5	12.0	24.8	20.1	7.5
10		7.0	6.3	19.7	15.4	6.2
14		4.0	3.0	12.3	11.3	4.7
20		2.7	2.1		7.3	4.7
28		1.7	1.7		4.7	5.6
35		1.5	1.5		3.6	7.1
48		0.8	1.3		3.0	8.3
65		0.4	0.9		1.9	6.9
100		0.4	0.9		1.5	6.8
150		0.2	0.7		1.1	5.6
200		0.2	0.6		0.6	3.7
−200		0.6	2.1		3.0	21.8
	100.0	100.0	100.0	100.0	100.0	100.0
Reduction ratio by mean mesh of chert = 2.03 Reduction ratio by mean mesh of dolomite = 2.28				Reduction ratio by mean mesh of chert = 1.73 Reduction ratio by mean mesh of dolomite = 6.75		

STAGE GRINDING

Stage grinding has been talked about a great deal, but the subject has been neglected in the literature. The failure to put it into practice is outstanding. It is true that unusually heavy circulating loads have been attempted, but a study of grain sizes has not received enough scientific attention. Stage grinding has almost unlimited physical possibilities, and an examination should be made to see what the limit is and where the practice fails.

In the early part of the laboratory work on closed-circuit ball milling, finished products were made which contained as much as 72 per cent of minus 200-mesh size. These results were probably as good as average commercial practice. Later work was improved, so that for the same finishing size the amount of minus 200-mesh was reduced to only 23 per cent. Thus, the amount of ore finished at 48 mesh was increased greatly, but no information was available to indicate how much more increase might be expected by still better stage grinding. To get this information a test was made with a set of rolls. After each pass through the rolls the sample was screened to remove the finished size, and the oversize was returned for another stage of gentle grinding. The rolls were set slightly closer for successive passes, and about 40 passes were required. Finally, the finished product was screened and found to contain only 22 per cent of the minus 200-mesh size; what is more important, over half of the sample was on the 65-mesh sieve. The sizing analyses of these three products of

TABLE 9.—*Sizing Analyses of Products from Grinds of Three Degrees of Stage Grinding*

Size Mesh	New Feed, Per Cent	Finished Product, Per Cent		
		Poor	Fair	Good
6	5.6			
8	8.4			
10	8.3			
14	6.8			
20	6.2			
28	6.1			
35	18.9			
48	20.3	0.6	3.2	
65	11.3	0.7	20.1	56.0
100	6.3	2.7	35.8	11.8
150	1.0	9.6	13.3	6.3
200	0.3	14.2	4.3	3.5
−200	0.5	72.2	23.3	22.4
	100.0	100.0	100.0	100.0

poor, fair, and good stage grinding are given in Table 9. No doubt it is physically possible for a ball mill to reproduce the results achieved by the rolls, but to do so would require a short mill, a large circulating load and unusually thorough classification.

Stage grinding may be evaluated in many ways. It is commonly expressed as percentage of circulating load, a figure that tells the relative tonnage of material that passes through the mill more than once. This value, in the light of what has been said regarding the importance of removing finished material, the advantage of increasing feed rate, and the rationing of ball and grain sizes, is obviously insufficient for comprehensive understanding of stage grinding. Several other critera could be used; the more important are: (1) useful circulating load; (2) tons per horsepower-hour; (3) reduction ratio; (4) percentage increase in surface units; (5) relative grain size of circulating load.

In the discussion of the first four of these, reference will be made to the data from an extensive series of tests on a 6½ by 12-ft. ball mill. More will be said of these particular tests later, but for this analysis the 36 tests were condensed into 12 groups, each representing a particular combination of operating conditions. The data are presented in Table 10.

Useful Circulating Load

The ordinary calculation of circulating load gives the same credit for the overgrinding of already finished material as it does for the grinding of oversize. The calculation of *useful* circulating load, however, considers the material accumulated on each sieve in the sizes above the finishing

TABLE 10.—*Criteria in Stage Grinding Illustrated by Results of* 36 *Tests on a* 6½ *by* 12-*ft. Ball Mill*
Arranged in 12 Groups

Size Mesh	Group A	Group B	Group C	Group D	Group E	Group F	Group G	Group H	Group I	Group J	Group K	Group L
USEFUL CIRCULATING LOAD, CUMULATIVE PER CENT												
3 to 6	130	68	80	33	43	64	55	53	9	26	130	190
On 8	83	27	29	28	30	33	36	19	9	18	74	88
On 10	85	33	34	33	35	36	45	21	12	20	80	88
On 14	92	36	41	40	43	38	51	25	14	25	93	100
On 20	105	48	52	50	60	51	66	32	20	33	110	110
TON PER GROSS HORSEPOWER-HOUR												
	0.39	0.27	0.28	0.29	0.33	0.27	0.32	0.22	0.21	0.24	0.42	0.46
REDUCTION RATIO BY MEAN MESH IN ONE PASS												
	2.52 to 1	3.72 to 1	3.33 to 1	3.24 to 1	3.17 to 1	3.65 to 1	3.25 to 1	4.13 to 1	5.70 to 1	3.95 to 1	2.19 to 1	2.20 to 1
PERCENTAGE INCREASE IN SURFACE UNITS (200 MESH)												
	210	310	290	270	270	310	300	330	450	320	190	190

size. These values are presented in the upper part of Table 10, where the calculation is carried down to 20 mesh. The calculation could not be carried further because the finishing size was 28 mesh.

In the tests the composite ball-mill feed and the discharge were sampled; the calculation for circulating load comprised: (1) subtracting the cumulative percentage on a given screen in the discharge from the corresponding percentage in the feed (the amount ground out), (2) dividing this value into the percentage in the discharge (the amount returned), and (3) multiplying by 100 to give percentage of useful circulating load.

The table shows that the circulating load is built up slightly more on 6 mesh than on 8 mesh. From 8 mesh the amount increases as the mesh approaches the finishing size, indicating a healthy grinding condition and a proper rationing of ball size. The building up on 6 mesh indicates that the balls are not too large, and the smallness of the actual amount of 6-mesh size shows that the balls are not too small. The crowding near the finished size is desirable because the nearly finished material should receive the best stage grinding. In this instance 2-in. balls were used. They are rationed to the smaller size but are large enough to prevent the 6-mesh grains from overcrowding the circulating load.

It might seem that a still larger over-all circulating load could have been used to advantage; but here an operating obstacle was met. In group L the composite feed was 100 tons per hour, the limit of the carrying capacity of the auxiliary equipment.

Tons per Horsepower-hour

The accord between tons per horsepower-hour and useful circulating load is obvious in Table 10. The better the stage grinding the higher are both of these values, but tons per horsepower-hour may be used as a yardstick in measuring stage grinding irrespective of the type of circuit. To illustrate this and to give a comparative view of amount of staging accomplished in ball mills and other devices, a digression will be made from ball milling to consider this value as applied to crushing machinery. Some examples of the tons treated per horsepower-hour by rolls in the same district are shown in Table 11. As compared with the value of 0.46 ton per horsepower-hour for the ball mill in group L, Table 10, the general trend in practice is for the tons per horsepower-hour to be increased in the coarse-size crushing. Technically this is illogical, for it has been shown that as the finishing size is approached stage grinding should be increased. That is, from a technological standpoint the coarse material should be treated very severely, while the nearly finished material should be gently staged to avoid overgrinding. If crushers could be built to give a still higher reduction ratio, the advantage would be both technical and practical.

TABLE 11.—*Data on Rolls Crushing Dolomite*

Plant	Rolls		R.p.m.	Initial Size, In.	Finishing Size, Mesh	Tons per Hp-hr. (Gross)	Efficiency, Surface Tons per Hp-hr. (65 Mesh)
	No.	Size, In.					
C		42 by 24	63	2.08	6	2.00	24.0
D		36 by 15		1.05	8	1.13	24.0
E	3	42 by 18	75	0.75	4	1.00	15.4
	3	42 by 18	75	0.75	4	1.28	16.5
	4	42 by 18	70	0.75	4	2.10	21.1
	4	42 by 18	70	0.75	4	2.54	23.7
	5	42 by 18	90	0.75	4	2.15	14.3
	5	42 by 18	90	0.75	4	2.24	17.1
	6	42 by 18	64	0.75	4	1.71	24.3
	6	42 by 18	64	0.75	4	2.00	27.9
	5[a]	42 by 18	90	0.75	4	0.93	17.9
F		42 by 18	68	1.05	8	1.63	19.3

[a] Rolls set excessively close.

To view stage crushing from another angle, tons per horsepower-hour are translated here into a theoretical number of feet of drop. Severe treatment is equivalent to a long drop and gentle treatment to a short drop. The extremes in grinding practice are illustrated strikingly by using this value.

Probably the record for extreme stage grinding is a test used in determining the "friability" of coal. The coal is dropped from a height of 10 ft. on to a hard floor, and the reduction is measured. Translating 10 ft. of drop (neglecting friction) into tons per horsepower-hour gives approximately 100 tons per horsepower-hour. This mild treatment has been considered by some as a test for "friability" rather than "grindability."

It is not the purpose of this paper to inquire where friability ends and grindability begins in the discussion of a grinding treatment that may be extremely gentle or extremely drastic, but to show the wide range of stage grinding met. For coal the drop is 10 ft. By the same yardstick the equivalent drop of the flint in the tests recorded here was one mile.

The Hadsel mill gives a high degree of stage grinding.[11] The actual drop through the air is approximately 20 ft. This is equivalent to a high feed rate with respect to the power—50 tons per horsepower-hour. By Table 10, where high circulating load corresponds to high tons per horsepower-hour, the circulating load of the Hadsel mill would be expected to be high. By use of the rate of *new* feed (8 tons per hour) and *net* power (approximately 56 hp.), it is found that the total tons per hour in the Hadsel mill is 2800 and the circulating load 35,000 per cent. Table 12 gives a record of some examples of tons per horsepower-hour and the equivalent height of theoretical drop.

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[11] J. B. Huttl: Gravity Milling and Flotation of Low-grade Gold Ores. *Eng. & Min. Jnl.* (1932) **133**, 631.

Reduction Ratio by Mean Mesh

A high reduction ratio in one pass as determined by mean mesh indicates drastic treatment and corresponds to a low value of stage grinding. Thus reduction ratio is inversely indicative of tons per horsepower-hour as shown in Table 10, where also it will be seen that it varies inversely as the circulating load.

Percentage Increase in Surface Units

Like reduction ratio, the percentage increase in surface units varies inversely as the stage grinding. Theoretically, a reduction ratio of 2 to 1 corresponds to 100 per cent of new surface. Actually the values fail to correspond, as in Table 10, because the surface units are figured as useful grinding to 200 mesh and the reduction ratio is computed by a short-cut method in which "mean mesh" is substituted for the exhaustive method involved where average diameters are calculated.

TABLE 12.—*Examples of Tons per Horsepower-hour Converted to Equivalent Feet of Drop*

Tons per Horsepower-hour		Drop, Ft.
100	Coal friability	10
50	Hadsel	20
10		100
2	Rolls, Table 11	500
1	Rolls, Table 11	1000
0.6	Laboratory dolomite	1670
0.46	Group L, Table 10	2170
0.20	Laboratory flint	5000

Relative Grain Size in Circulating Load

Since good stage grinding calls for a small reduction ratio in any one pass, the grain size can be used as a criterion of the staging, but it may also be used in the study of the operating conditions. Unfortunately, the grain size of the circulating material has not received in practice the consideration it should have. To illustrate this, two examples have been taken from a summary of fine grinding in commercial plants.[12] These examples are cited without criticism of the actual practice, for other conditions may justify the operation. They will be discussed purely from a ball-milling viewpoint. One example is shown by the data in Table 13.

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[12] A. M. Gow, M. Guggenheim and W. H. Coghill: Review of Fine Grinding in Ore Concentrators. U. S. Bur. Mines *Inf. Circ.* (in press).

In this case the mill was loaded to crush coarse ore, but the feed rate was so small and the required over-all reduction ratio so great that before the ore reached the discharge all the coarse feed was reduced and only fine sand was left for the coarse-grinding media to combat. After the finished material was removed from the discharged pulp only fine sand remained to be sent back as a circulating load. Probably the operator rejoiced to see that the mill had reduced all the coarse ore, but he overlooked what should cause concern: coarse-grinding media are inefficient in grinding fine sand (see Table 4). The coarsest size in the feed is 1.05 in., and the finishing is at 65 mesh. No human hand will ever load a ball mill with a grinding medium that will work efficiently through such a broad range. The practice looks like a hangover from the period when ball mills were pictured as without limitations.

TABLE 13.—*Ball-mill Products When Required Reduction Ratio Was Great and Balls Were Too Large*

Size	Weight, Per Cent				
	New Feed	Composite Feed	Mill Discharge	Circulating Load	Finished Product
Inches					
1.05	7.4	1.7			
0.742	10.8	2.5			
0.525	14.0	3.2			
0.371	12.9	3.0			
Mesh					
3	9.9	2.3			
4	7.8	1.8			
6	6.2	1.4			
8	5.4	1.2			
10	3.9	2.7	2.2	2.4	
14	3.1	2.0	1.6	1.7	
20	2.5	2.8	3.0	2.9	
28	2.7	5.3	7.2	6.1	
35	2.5	8.8	10.0	10.6	
48	1.8	17.0	15.1	21.6	
65	1.9	17.4	13.3	22.1	2.0
100	2.0	10.0	12.1	12.3	8.9
150	1.7	5.1	5.9	6.1	7.9
200	1.2	3.4	4.4	4.0	7.9
−200	2.3	8.4	25.2	10.2	73.3
	100.0	100.0	100.0	100.0	100.0
Ratio, tons	100	435	435	335	100

If a figure on circulating load, per se, has any significance, the fact that it is 335 per cent would have to be accepted as indicating good stage grinding; but examination shows that one-third of the circulating load is already finished, and all of it is through 8 mesh. This is mixed with a new feed that is almost 75 per cent coarser than 8 mesh. Such a mixture prohibits both good stage grinding and efficient operation. It seems that better grinding practice would have resulted if the feed had been roll-crushed through 4 mesh before ball milling. As corroborative evidence the relatively high efficiency of rolls is shown in Table 11.

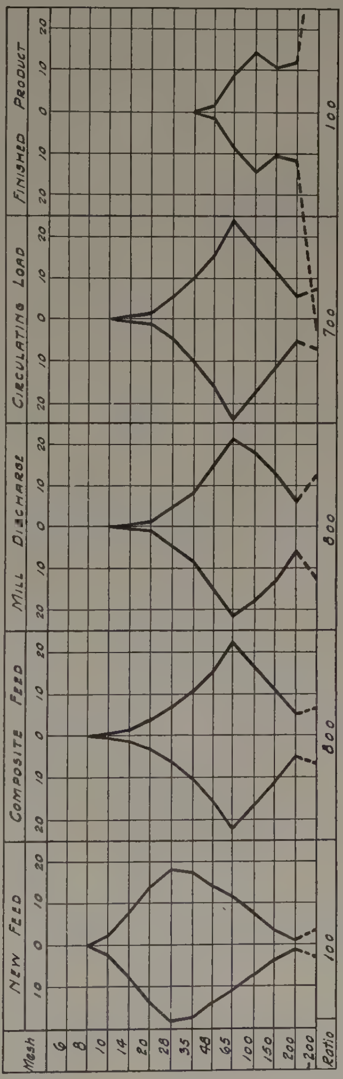

Fig. 15.—Sizing diagrams of ball-mill products in a western plant when reduction ratio was moderate and ball size was well chosen.

If heterogeneity of the ore accounted for the wide spread in sizes, the argument for rolls is the more pertinent, for it is impossible to stage-grind a heterogeneous ore in a ball mill.

A plant example that comes more nearly illustrating good stage grinding is represented by the data shown in Table 14 and Fig. 15. Here the new feed and circulating load have sizing analyses that are within the same range of sizes, so that a ball charge that must always be rationed to the new feed would be quite efficient on the circulating load. This is shown by the similarity of the diagrams in Fig. 15, which may be compared in spite of the fact that the products are all burdened with slime.

The similarity of the diagrams would suggest that the balls, which in this operation are 2½-in. maximum, are about the correct size. This can be investigated further by calculating the useful circulating loads, as has been done in Table 15, and similarly done in Table 10. The calculation in Table 15 is carried only to 48 mesh because finer sizes are not wholly returned. The useful circulating load is 59 per cent in the coarsest size and increases regularly to the over-all useful circulating load of 312

TABLE 14.—*Ball-mill Products When Reduction Ratio Was Moderate and Ball Size Well Chosen*

Size, Mesh	Weight, Per Cent				
	New Feed	Composite Feed	Mill Discharge	Circulating Load	Finished Product
10	2.2	0.3			
14	7.4	1.6	0.5	0.8	
20	14.1	3.4	1.6	1.8	
28	18.2	6.8	4.3	5.1	
35	17.6	10.8	8.1	9.8	
48	13.9	15.2	14.6	15.4	1.7
65	11.6	22.4	21.6	24.0	8.6
100	7.3	16.8	18.0	18.1	14.0
150	3.4	11.4	12.9	12.6	10.5
200	1.2	4.9	6.1	5.5	11.8
−200	3.1	6.4	12.3	6.9	53.4
	100.0	100.0	100.0	100.0	100.0
Ratio, tons	100	800	800	700	100

per cent as compared with the commonly accepted figure of 700 per cent for the ordinary circulating-load calculations. The facts that as much as one-half of the weight of the useful circulating load is concentrated between the 35 and 48-mesh sieves, and that there is only a very small load in the coarsest size, indicate that 2-in. balls would do better work than the present 2½-in. ones. This change of ball size might also aid the classification, since the coarser sand would build up slightly; it is difficult to carry on desliming in the presence of much fine sand.

TABLE 15.—*Consideration of Useful Circulating Load from Data in Table* 14

Size, Mesh	Relative Weights, Sieve by Sieve,[a] Per Cent				Useful Circulating Load, Cumulative Per Cent
	New Feed	Composite Feed	Mill Discharge	Useful Circulating Load, Weight	
14	9.6	15.2	4.0	5.6	59
20	14.1	27.2	12.8	12.6	77
28	18.3	54.4	34.4	35.7	130
35	17.6	86.4	64.8	68.6	207
48	13.9	121.6	116.8	107.1	312
Total......	73.4	304.8	232.8	229.6	312

[a] The failure of the quantities to be in complete accord is due to small errors in screen analyses.

REGULATION OF COMMERCIAL MILLS

If a change in the operating conditions of a commercial ball mill is advocated it generally is either to increase the grinding capacity for additional feed or else to reduce the grinding capacity and power to accord with a small amount of set feed. The field tests to confirm laboratory observations have encountered both of these conditions.

Tests to Increase Capacity

In one plant it was desirable to find the conditions under which a 6½ by 12-ft. ball mill (converted from a rod mill) should be operated to give the maximum capacity. Thirty-six grinding tests were made over a period of about two months, at various mill volumes, speeds and feed rates. The ball charges were 41, 45, 50, 55, and 59 per cent of the mill volume. The drive pinions were changed to give speeds of 52, 60 and 67 per cent of the critical, and the feed rates were 38.2 to 101 tons per hour, usually about 50 tons per hour. The ore was dolomitic. The similarity of some of the 36 tests permitted combining them by averages into 12 groups. The data from these combinations are given in Fig. 16.

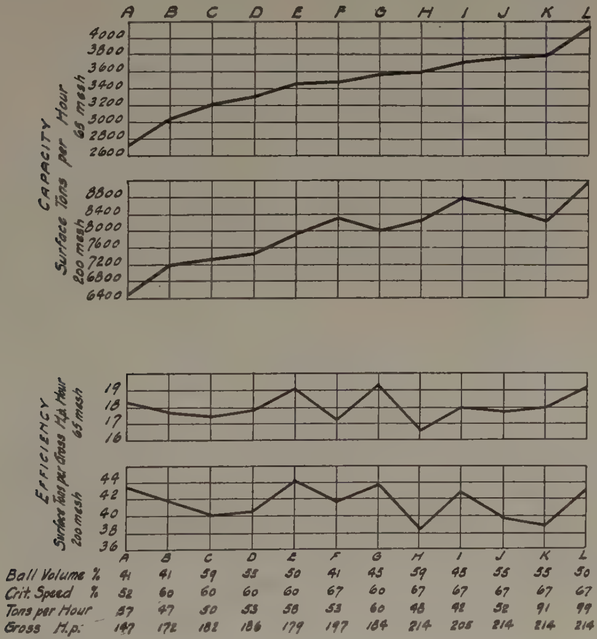

FIG. 16.—CAPACITY AND EFFICIENCY VALUES AND OPERATING DATA FROM 36 TESTS ON A 6½ BY 12-FT. BALL MILL. TESTS GROUPED, AND ARRANGED BY ASCENDING 65-MESH CAPACITY VALUES.

These are the tests shown in Table 10. The data sheet from one of the tests in group L is shown in Table 16, and the sieve analyses from this test are portrayed in Fig. 17. Data sheets were made on each one of the 36 tests.

TABLE 16.—*Data Sheet from One Test in Group* L, *Figure* 16

Size Mesh	Mean Mesh (M)	Mean Surface (S)	Feed			Discharge		
			Weight, Per Cent (W)	W × M	W × S	Weight, Per Cent (W)	W × M	W × S
Inches								
1.05	1	1						
0.742	2	1.41						
0.525	3	2						
0.371	4	2.82						
Mesh								
3	5	4						
4	6	5.64	0.4	2.4	2.3	0.2	1.2	1.1
6	7	8	1.7	11.9	13.6	1.1	7.7	8.8
8	8	11.28	12.0	96.0	135.4	4.9	39.2	55.3
10	9	16	15.2	136.8	243.2	6.7	60.3	107.2
14	10	22.56	14.4	144.0	324.9	7.9	79.0	178.2
20	11	32	13.0	143.0	416.0	8.9	97.9	284.8
28	12	45.12	15.6	187.2	703.9	13.2	158.4	595.6
35	13	64	11.7	152.1	748.8	12.7	165.1	812.8
48	14	90.24	6.3	88.2	568.5	9.4	131.6	848.3
65	15	128	4.9	73.5	627.2	8.9	133.5	1139.2
100	16	180.48	2.7	43.2	487.3	6.2	99.2	1119.0
150	17	256	1.1	18.7	281.6	4.2	71.4	1075.2
200	18	360.96	0.5	9.0	180.5	3.2	57.6	1155.1
−200	19	512	0.5	9.5	256.0	12.5	237.5	6400.0
Totals........			100.0	1115.5	4989.2	100.0	1339.6	13,780.6

Tons per 24 hours....................	2430	Mean surface of feed.....	49.89
Tons per hour........................	101.25	Mean surface of discharge.	137.81 (−65-mesh)
Per cent solids......................	78.0	Units of surface reduction.	87.92 (40.92)
Horsepower...........................	211.9	Surface tons per hour.....	8,902 (4143)
Tons per hp-hr.......................	0.478	Surface tons per hp-hr....	42.03 (19.56)
Mean mesh of feed, mm..............	0.790	Ball load, lb.............	55,000
Mean mesh of discharge, mm.........	0.363	R. p. m. of mill..........	21.13
Reduction ratio.....................	2.18	Type of mill, 6½ by 12 ft., alternating current	

The calculations of *capacity* and *efficiency* are made at both 200 mesh and 65 mesh (the figures in parentheses in Table 16). In the graphs of Fig. 16 the groups are arranged, from left to right, in ascending order of 65-mesh capacity values. In this compilation the five groups of tests with highest capacity are L, K, J, I and H. These were all run at the highest speed and had the greatest power-consumption values.

The five groups of highest efficiency are G, E, L, A and I, and these include all the tests at mill volumes of 45 and 50 per cent and the slow-speed runs at 41 per cent mill volume. The groups of low efficiency are those of 55 and 59 per cent mill volume. However, the deviation from the mean is only 8 per cent, so that the grinding is nearly proportional to the power.

As to the ultimate capacity, a power increase of about 20 to 25 per cent could be expected by increasing the speed to 80 per cent of the critical (Fig. 3). In order that maximum grinding might be obtained, the feed rate should also be increased proportionally. However, in this instance, that would not be feasible, so the estimate is probably a little high. To maintain efficiency the ball charge should not occupy more than 50 per cent of the mill volume.

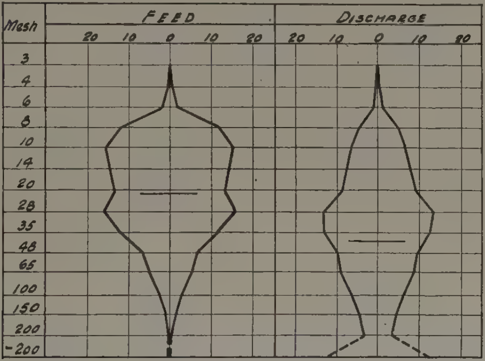

FIG. 17.—TYPICAL SIZING DIAGRAMS OF FEED AND DISCHARGE FROM TEST ON 6½ BY 12-FT. BALL MILL. (SEE TABLE 16.)

In connection with this test, although the concentrator was stressing capacity rather than metallurgy, the composite feed is worthy of attention. The grinding circuit is closed by a hydraulic classifier and tables, accounting for the unusually thorough desliming of the actual feed to the mill. Compare the composite feed diagram in Fig. 17 with that in Fig. 15.

Tests to Increase Efficiency

In another plant the problem was to increase the mill's efficiency by reducing power, for the mill was doing more grinding than was needed. This was indicated by the absence of coarse material in the discharge.

The mill was loaded to 50 per cent of the mill volume with 3-in. maximum-size balls and was belt-driven at 80 per cent of the critical speed. It was a 6 by 4-ft. mill, of overflow type, with a 9-in. discharge opening. The ore was dolomitic, and the circuit was closed by a vibrating screen. By repeated changes of the drive pulley on the motor, slower ball-mill speeds were obtained. The changes were gradual, and in all five pulleys were used. Each speed was observed for a week, during which testing was done. Ultimately, the speed was reduced to 60 per cent of the critical, and the gross power about one-third. The data showing the progress of these tests are in Table 17. Gross power was obtained by a recording wattmeter, and net power was calculated from the motor characteristics and an actual dead load test. The metallurgical recoveries in the succeeding steps of concentration were not impaired by the results of this power saving.

TABLE 17.—*Results of Reducing Speed of* 6 *by* 4-*foot Mill When Amount of New Feed and Other Operating Conditions Were Constant*

Test	Ball Volume, Per Cent	Speed, Per Cent Critical	Power		Feed, Tons per Hour	Surface Tons of 65-mesh	
			Gross Kw.	Net Hp.		Per Hour	Per Net Hp-hr.
A	50	80	65	67	13.1	1278	19.1
B	50	72	58	60	13.3	1268	21.1
C	50	69	55	56	12.6	1220	21.7
D	50	63	48	49	17.3	1300	26.5
E	50	60	45	46	18.8	1246	27.3

The total amount of grinding, as measured by surface tons per hour (65 mesh), was about the same in all tests. This is what was to be expected because, as was stated above, there was a set amount to be ground. The economy was in the saving of power and, incidentally, in the saving of ball wear through reduced speed.

Another method of accomplishing the same end was used on a similar mill in an adjacent section of the same plant. Here the speed was left the same, and the power and grinding were reduced by using a smaller ball charge. The data from tests at 50, 37 and 31 per cent ball volume are shown in Table 18. Decreasing the ball volume increased the efficiency by using less power to do the required amount of grinding.

Certain tests may be cited to compare low-speed and high-mill-volume grinding with high-speed and low-mill-volume grinding. These are grouped in Table 19, which shows also the sizing analyses of the mill discharges. The striking similarity of the products of tests C and F and of tests E and G is added evidence that impact and attrition refer to degrees of grinding rather than to types. The advantage of G over E is due to the higher feed rate.

TABLE 18.—*Results of Reducing Ball Volume of* 6 *by* 4-*foot Mill When Amount of New Feed and Other Operating Conditions Were Constant*

Test	Ball Volume, Per Cent	Speed, Per Cent Critical	Power		Feed, Tons per Hour	Surface Tons of 65-mesh	
			Gross Kw.	Net Hp.		Per Hour	Per Net Hp-hr.
A	50	80	65	67	13.1	1278	19.1
F	37	79	58	54	12.9	1271	23.3
G	31	79	51	46	21.1	1340	28.4

TABLE 19.—*High-speed Low-volume Grinding Compared with Low-speed, High-volume Grinding in 6 by 4-foot Mills*

Test	Ball Volume, Per Cent	Speed, Per Cent Critical	Power		Feed, Tons per Hour	Surface Tons (65-mesh)	
			Gross Kw.	Net Hp.		Per Hour	Per Net Hp-hr.
C	50	69	55	56	12.6	1220	21.7
F	37	79	58	54	12.9	1271	23.3
E	50	60	45	46	18.8	1246	27.3
G	31	79	51	46	21.1	1340	28.4

SIZING ANALYSES OF DISCHARGES

Size, Mesh	Weight, Per Cent			
	Test C	Test F	Test E	Test G
6			0.1	0.1
8	0.4	0.4	1.9	1.5
10	1.5	1.1	4.7	3.7
14	3.9	2.8	9.0	8.4
20	6.0	5.3	11.4	12.6
28	6.9	7.1	10.4	11.5
35	8.8	9.5	10.9	11.5
48	7.6	8.5	7.8	7.7
65	7.6	8.3	6.3	6.4
100	7.2	7.5	5.3	5.4
150	6.9	7.2	4.7	4.2
200	6.2	6.4	4.0	4.2
−200	37.0	35.9	23.5	22.1
	100.0	100.0	100.0	100.0

Ball volumes less than 31 per cent were tried but were unsatisfactory. Probably the failure was due to an excessive amount of ore for the small volume of balls.

Both methods reduce the overgrinding and tend to give larger circulating loads with better attendant stage grinding.

SUMMARY AND CONCLUSIONS

A new philosophy has been developed in the science of ball milling by using power as an index of the ball action and by ascertaining the effects and the interdependence of the many variables of operation, which have been segregated into two classes, set and induced. The research has revealed certain underlying principles that have puzzled earlier investigators. It has shown that a study of ball paths and trajectories cannot be expected to explain the power requirements or grinding capacity of a mill; that no set variable may be studied comprehensively without considering the associated induced variables; that impact and attrition differ only in amount and degree of grinding, not in type; that the percentage of over-all circulating load is not necessarily indicative of the amount of stage grinding and that *useful* circulating load data should be used to give a true analysis; that rationing of feed rate to ball action (power) and of ball size to grain size are necessary for good operation; that the proper ball size is independent of the hardness of the ores tested; and that the grinding may be made proportional to the power expended.

The power study has shown how the variables, including slipping and the amount of ore in the mill, affect the power; and a method and formula are offered whereby the power required by a cylindrical mill of any size and operating under any conditions may be estimated. This also makes possible correlation of the performance of various commercial mills.

The effects on power and grinding of the following are discussed: The size and design of the mill; the amount and size of the ball charge; the size, type, feed rate and pulp density of the feed; the speed of rotation of the mill; the efficiency of classification in closed circuit; slipping; and the amount of material in the mill at any instant.

Several means for measuring the degree of stage grinding, both in open and closed circuits, have been offered, and a suggestion has been made regarding the amount of circulating load for good stage grinding. The necessity for thorough classification is pointed out, and an example is given to show the harm done by allowing the feed to carry material of finished size. In this connection it is suggested that the present closed circuits be supplemented by hydraulic classifiers and tables to avoid differential and overgrinding and that rolls might be used advantageously in the preparation of finer feed for ball mills so that the reduction ratio in the final stage of grinding need not be so great. The efficacy of hydraulic classifiers and tables for desliming and the efficiency of rolls in fine grinding are illustrated.

Laboratory tests were made with a 2 by 2-ft. mill and with extremely hard and soft ores, which were of the same grain size and were obtained in carload lots from the Tri-State district and the Southeast Missouri lead belt. Field testing was carried on in both these districts.

Examples of the practical application of the principles of ball milling are found in the correlation of the laboratory and field testing. One set of commercial mills was adjusted to increase the grinding capacity; and in another plant, where the mills were found to be overgrinding, their operation was changed to make them more efficient. The several methods for accomplishing both these ends have been discussed.

References to the articles on ball milling by other authors have been omitted because justice cannot be done to the vast amount of literature without lengthy digression from the subject of this paper. They will be reviewed in connection with a paper on the history of fine grinding now in preparation.

The need for further research on the amount of ore in the mill—one of the induced variables—has been made evident, and a continuation of this general investigation will assume that course.

The next phase of the fine-grinding problem to receive attention will be rod milling. The laboratory set-up is shown in Fig. 18, and the investigation will follow in the wake of this ball-mill study. In addition, a set of "frictionless" rolls has been built by replacing the usual babbitt bearings on a pair of 6 by 10-in. rolls with roller main bearings and ball thrust bearings. The set-up is equipped with a recording wattmeter; and tests for grinding capacity and grinding efficiency will be made with the feed used in this ball-mill investigation.

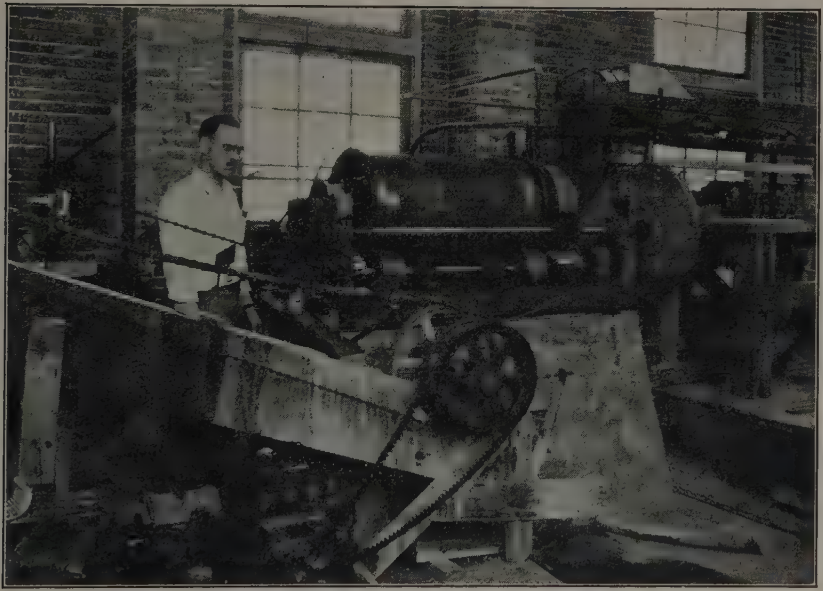

FIG. 18.—LABORATORY ROD-MILL SET-UP.

DISCUSSION

(Charles E. Locke presiding)

C. E. LOCKE,* Cambridge, Mass.—The paper states it to be definitely settled that results from laboratory mills are applicable to large-scale mills. Would the author tell us how he satisfied himself on this score?

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* Professor of Mining and Ore Dressing, Massachusetts Institute of Technology.

W. H. Coghill.—The statement that Professor Locke repeated may not be literally true, but we can adjust one of the small mills to give the same results as the big ones. It has been hard for me to understand how a little mill can exercise the forces necessary to exceed the elastic limit of the grains of feed. It does. In mills much smaller than the one we are using, the ore is cracked, and when the tons per horsepower-hour are taken into account, the results with this little mill are parallel with those of the 6 by 12-ft. mill.

H. W. Newton,* New York N.Y.—Has this work been done in overflow types of mills or with low-level types?

W. H. Coghill.—Overflow.

Member.—What is the effect of a high-specific-gravity ball in the load upon its grinding?

W. H. Coghill.—We have what we think is a very complete little report on that very thing, in a paper to be published by the Bureau of Mines.[14]

V. E. Flanagan,† Dorchester, Mass.—Mr. Coghill spoke of the breakage of materials due to dropping. The Bradford breaker works on this principle. It is generally 10 to 12 ft. in diameter. It lifts the coal and drops it through a screen, and the coal is crushed. Several years ago in the St. Louis district I was selling machinery. I visited a barite plant and took some samples back to the office in St. Louis. I dropped one of the samples on the marble floor at the entrance to the office and it broke into several fragments. In laying out that plant they wanted to produce as few fines as possible. I persuaded the manager to put in a modified Bradford breaker. It worked beautifully and I think saved him a lot of money and produced less fines. I think sometimes we do too much grinding.

W. H. Newton.—Mr. Coghill stated that similar results were obtained in a 2-ft mill. Were any tests run to determine whether differences in the ratio of length to diameter made any difference?

W. H. Coghill.—Length is taken into account in the formula for calculating the power required to run a big mill, on page 39.

W. H. Newton.—I mean the curve.

W. H. Coghill.—That is on page 30.

B. R. Bates, Jackson Heights, N.Y.—Is your work going to include the use of ore for grinding; for example, South African practice?

W. H. Coghill.—We have not used that particular material. The supplementary paper[15] will come very nearly to answering your question.

B. R. Bates.—You are no doubt familiar with the South African practice.

W. H. Coghill.—Doubtless it cuts down the capacity tremendously.

B. R. Bates.—When it comes down to the cost of balls, it is another question.

W. H. Coghill.—If you reduce the specific gravity, you reduce the capacity.

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* New York Manager, The Mine & Smelter Supply Co.

[14] W. H. Coghill and F. D. De Vaney: Conducting Grinding Tests for Easy Interpretation of Results. U. S. Bur. Mines *Rept. of Investigations* 3239 (1934) 3–8.

† Plant Engineer, Walter Baker & Co., Inc.

[15] Reference of footnote 14.

C. Legrand,* Douglas, Ariz. (written discussion).—To me the definition of critical speed given in footnote 2 is a little indefinite, as this speed would be variable according to size of balls. Is it intended to be the speed at which the centrifugal force at internal radius of lining is equal to gravity?

One important element missing in the investigation, which I know is difficult to obtain, but is of great importance commercially, is the wear of balls and lining per surface ton under variable conditions of load and speed. Have the authors any information on this subject?

M. W. von Bernewitz, Sydney, Australia (written discussion).—This paper reminded me of my paper[16] written in 1911, and of my recent observations on ball mills, tube mills and rolls in Australia and New Zealand. Of course, I have watched ball mills and tube mills working in California, Colorado, Nevada, Utah, Ontario and Quebec.

At the time I covered ball milling in the Western Australia gold center, all of the Krupp ball mills were crushing ore dry for subsequent roasting. Tube mills were working in the concentrating plants. Now, a few dry ball mills are operated in three plants (Associated, Great Boulder, South Kalgurli); the second named has since been supplanted by a plant with wet ball mills; and two plants (Boulder Perseverance and Lake View & Star) have wet ball mills; one with tube mills, the other without them. At two plants (the Associated and Associated Northern) where I had some authority I paid considerable attention to ball-mill speed, ball loading and discarding very small balls, ore feeding, sound of mill, liners and screens. These mills had peripheral discharge and did good work on hard ore. Unlike the writers of the paper under discussion and Haultain in Ontario, I was unable to look squarely into the side of a ball mill, but by means of a portable electric lamp, placed well down in the mill-feed hopper, the action of the balls and ore could be plainly seen through the feed nave, particularly when some rather damp oxidized ore was being crushed. There was no trajectory effect. The balls were carried up nearly level with the mill center line and then rolled to the bottom. I was satisfied that impact has little to do with crushing, the action of the balls being one of rolling, grinding and abrasion. Of course, there is considerable attrition between the pieces of ore themselves. Photomicrographs of 30-mesh and 100-mesh products revealed the former to be somewhat rounded and the latter to have sharp, ragged edges, showing that the final particles are split by the rolling of the heavy balls. My paper is replete with all ball-mill data, perhaps applicable to Kalgoorlie practice until recent years.

With regard to circulating load (always astonishing to me) and classification and stage grinding, as discussed by Gow, Guggenheim, Campbell, and Coghill, at Kalgoorlie may be seen two opposite practices: The Boulder Perseverance has one Hardinge mill (another since added to increase plant capacity) in closed circuit with a Dorr classifier—one stage—this yielding the desired cyanidation product; the Lake View & Star has Australian ball mills in open circuit and two sets of tube mills in closed circuit with classifiers—three stages. The final product is finer than that of the Boulder Perseverance, but the Lake View & Star cyanides only its concentrates. At Broken Hill, New South Wales, the great lead-silver-zinc district, all four plants have rolls in circuit with vibrating screens; three plants (Central, South and Zinc) have tube mills in closed circuit with classifiers, and one plant (North) has Hardinge ball mills in closed circuit with classifiers.

As in the United States, there is in Australia a diversity of practice regarding ball sizes and between steel and flint pebbles. At Kalgoorlie, the Boulder Perseverance and Lake View & Star use 5-in. steel balls in their mills, and the latter uses Danish flints in its 22-ft. tube mills and 2-in. steel balls in its 11½-ft. tube mills. At Broken Hill, the North uses 2-in. cast balls in its ball mills, and the South uses this size in its tube mills; the Zinc company uses South Australian flints in its tube mills. At Portland, N.Z., Wilson's Portland Cement Co. uses 2, 3 and 5-in. steel balls in separate compartments in its Smidth wet mills, and two sizes of balls in its Smidth and Allis Chalmers dry mills. At Waihi, N.Z., the tube mills grinding gold-silver ore are half filled with Danish pebbles.

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* Mechanical Engineering Department, Phelps Dodge Corporation.

[16] M. W. von Bernewitz: Ball Mill Practice at Kalgoorlie. *Min. & Sci. Pr.* (July 15, 1911).

R. T. Hancock, London, England (written discussion).—The statement, in the summary, that impact and attrition differ only in amount and degree of crushing, and not in type, is neither correct nor in harmony with the manner in which the subject is treated in the general body of the paper. The erroneous theories that the authors so completely demolish are that high speed, or cataracting, yields a product of a different type from that yielded by slow speed, or cascading, and that in the former the reduction is by impact and in the latter by attrition, or abrasion. None the less, impact and attrition do yield products of entirely different types. At least since the appearance of Prof. A. M. Gaudin's paper[17] the marked difference between them is known to be due to the nonemployment of balls large enough to crush effectively the larger pieces in the feed, so that these are mainly reduced by abrasion, which yields fines from the corners rubbed off while they are being rounded, but no intermediate sizes. The result is a size-distribution along the Tyler-screen base scale which tends to show two highs instead of the single one that results from normal crushing. Gaudin's data, and others in the literature, indicate that the diameter of the smallest effective ball must be at least 10 times that of the largest particle in the feed. If the mill discharge of Table 5 for small balls is calculated, it shows the two-peak effect well, and this even persists into the circulating load, which would not have been the case if it had been expedient to set the classifier to divide its feed in the neighborhood of the low saddle at 20 mesh. Even as it is, the efficiency of the classifier on this feed is remarkable, while that on the large-ball discharge, although much lower, would be considered about the normal in plant performance. The use of a ration of 1¼-in. balls is indicated for the effective crushing of this feed. The finished product from the large balls is much better suited for subsequent table work, but their use caused the circulating load to build up to an extraordinary degree, and the relative inefficiency of performance here was probably due in part to overloading of the classifier. The double-peak effect is almost absent in Table 13, which would indicate that the ball size was suited to the feed, and it would have been useful if the ball size had been given instead of the incredible statement that it was too large. Classifier efficiency here was about 70 per cent.

The supreme importance of the proportioning by weight of the sand pulp to the ball load has at last received mention in the literature. One method of rating crushing efficiency (not the authors') shows highest values when the amount of sand is about equal to the normal void space among the balls, say 13 per cent of the weight of the ball charge. This is supported by the efficiency curves given for the 8-in. discharge opening, but with a 4-in. opening it was impossible to secure this proportion with the ball load used. Table 2, from which these curves are drawn, might have been substituted by screening analyses of representative feeds and products for the benefit of those who like to make their own calculations. The statement on page 35 that maximum power was attained when the ore was approximately sufficient to fill the interstices of the balls at rest is hardly borne out by the curves on the opposite page, where the maxima occur at half this quantity or less.

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[17] A. M. Gaudin: *Trans.* A.I.M.E. (1926) **73**, 253.

The rule followed in calculating the useful circulating loads of Table 15 appears to be: Subtract the total weight (not percentage) on the particular screen in the new feed from the total weight on the same screen in the composite feed, and divide the result by the total weight on that screen in the new feed. Or more simply, divide the total weights in the circulating load by those in the new feed. This is certainly not the rule given on page 61, and which one was followed in calculating the figures of Table 10 cannot be guessed at in the absence of screening analyses.

There seems to be an error in Table 14. If the mill was in simple circuit with a drag classifier, such figures as are given are impossible. The source of the error cannot be located with certainty by an outsider, but it seems to have been made in setting down the figures of the screening analysis of the mill discharge, where the amount stated as 12.9 per cent on 150 mesh should be approximately 7.6 per cent. The amount given as on 200 mesh is correct, but the amount through 200 mesh should be 17.6 instead of 12.3 per cent, to bring the total to 100 per cent. The corresponding figures for the circulating load and composite feed will be similarly affected, but nothing in Table 15, which does not extend to these fine sizes.

An expression for power of a more general form than that given by the authors may be suggested as

$$\text{Net horsepower absorbed by mobile load} = (0.00078)(C^3)(L)(\text{sp. gr.})(\text{r.p.m.})$$

where C is the length in feet from side to side of the mill of the chord defined by the surface of the total load at rest, L is the effective length of the mill in feet, and the other terms are self-explanatory. Slip is assumed negligible; if present it will proportionately reduce the value of the constant. The specific gravity of the load must take into account the grinding media, whether balls or pebbles, the ore charge, and its moisture.

The authors assume the dead load to be directly proportional to the speed. Dr. Geoffrey Martin[18] with his experimental set-up, obtained figures indicating that the dead load varied as the 1.5 power of the speed in revolutions per minute, while it was practically independent of the total load. If this is the general case, it might partly account for the peculiar shape of the curves of Fig. 3. In their earlier paper it was left to the reader to infer that the dead load was determined for each combination of speed and total load, while in this one the authors themselves make an inference from an untested hypothesis in a branch of mechanics where guessing is dangerous. They also seem to consider that the existence of a maximum in the power curve at about 85 per cent of the critical speed is due to the falling balls giving back some of their energy. But their earlier work showed this maximum at 65 per cent, and from the parabolic equation for ball path the outermost balls are not thrown halfway across the mill at this speed. In this earlier work, where slip was negligible, the power figures were quite accurately proportional to the 2.5 power of the diameter and to the speed, although the authors gave the exponent as 2.6 and made no mention of any relation to speed. In the present paper this straight-line relation to speed is stated to be characteristic of excessive slip, and that where there is none the line is curved. The section on slippage is the most difficult to understand, and it is not easy to follow the statement that a power curve made at the critical speed is low when slipping is small and high when there is much slipping, unless critical speed is not to be taken in its customary sense, but as that speed at which centrifuging actually begins. There is no mention at all of the effect of the angle of repose taken up by the load in the revolving mill under the influence of centrifugal force opposing gravity, which depends on the internal friction of the mass, a most complicated question, certainly, but one that cannot be neglected. The slow break of the power curves near the maximum as shown by the graphs cannot be accepted in the absence of figures. Determinations at intervals of 10 per cent of the critical speed are not close enough to decide this point against the earlier investigators.

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[18] G. Martin: *Trans.* Ceramic Soc. (1925–26) **25**.

W. H. Coghill.—Since reading Mr. Hancock's criticism. I have examined Table 14, and find that it is substantially correct. The data were taken from a paper by A. T. Tye (U.S. Bur. Mines *Inf. Circ.* 6261, on Milling Methods and Costs at the Concentrator of the Cananea Consolidated Copper Co., Cananea, Sonora, Mexico). Tye's figures check as closely as can be expected of mill tests. They were selected to illustrate a principle and not to show how accurate sampling could be performed in a mill. The principle that we were trying to put across was that "the new feed and circulating load have sizing analyses that are within the same range of sizes, so that a ball charge that must always be rationed to the new feed would be quite efficient on the circulating load." We had contrasted this condition with other places where a circulating load through 48 mesh was returned to enter the ball mill with 1-in. new feed.

Advantage of Ball (Rod) Mills of Larger Diameters and Advantage of Improving Bearings*

By Will H. Coghill,† Fred D. DeVaney,‡ and R. G. O'Meara,§ Members A.I.M.E.

The size of ball mills in the ore-dressing industry has increased from about 4 ft. in each dimension to 10.5 ft. in diameter by 8 ft. in length. In the cement industry they are as long as 45 ft. Plainly the trend has been toward larger mills. The common belief is that the big mills economize in floor space and first cost, but the advantage with respect to friction has not been clearly defined.

In the ball-mill studies at Rolla, Mo., laboratory mills as small as 2 by 2 ft. have been found as efficient in terms of the amount of grinding per unit of net power as mills 6 by 4 or 6 by 12 ft. Therefore, if supremacy can be claimed for the big mills, advantage must come from other sources. The advantage to be considered here is reduced loss by friction or dead load.

In the study to determine whether big mills have relatively less loss by friction the commercial catalogs were used for fundamental figures. The dimensions and weights of Table 1 are taken from the catalogs (the last line gives data on a Rolla laboratory mill). The net power—that is, the power expended within the mill for grinding—is calculated by a new formula.[1] The assumed speeds are each at 70 per cent of their theoretical critical speeds and the mills are loaded to the center with balls. In other words, they are assumed to be working under analogous conditions and are set for good grinding.

In the last column the ratio $B:A$ shows that the larger commercial mills are lighter per unit of net power than the smaller mills, and more of the motor output should therefore go into grinding. The laboratory mill seems to have some advantage over the larger mills; it would be expected to be light because it has no liners.

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[1] A. M. Gow, M. Guggenheim, A. B. Campbell and W. H. Coghill: Ball Milling. Page 39, this volume.

Another step must be considered. The number of revolutions that these mills make while each is running at 70 per cent of the critical speed must be taken into account. Table 2 includes the revolutions per minute; the calculations therein are based on the theory that the loss by friction varies as the weight and also as the speed (r.p.m.), since the mills are equally well built. By this premise the losses by friction vary approximately as weight times speed. Although this principle is not expected to hold through all speeds and weights, the manufacturers presumably would be at fault if they designed their bearings so that it did not hold through the working ranges.

TABLE 1.—*Relation of Total Weight to Net Power Required of Mills of Different Diameters and With Same Relative Ball Load and Speed*

70 PER CENT CRITICAL SPEED

Nominal Size of Mill, Ft.	Approximate Working Dimensions, Ft.	Net Horsepower by Formula (*A*)	Mill Weight,[a] Lb.	Balls, Mill ½ Full, Weight, Lb.	Total Weight, Lb. (*B*)	$\frac{B}{A}$
		Manufacturing Company No. 1				
3 by 5	2.6 by 5	8.4	12,500	4,000	16,500	1,960
5 by 5	4.6 by 5	35.1	33,000	12,400	45,400	1,290
6 by 5	5.5 by 5	55.0	47,000	17,500	64,500	1,170
7 by 5	6.5 by 5	83.6	66,000	25,000	91,000	1,090
8 by 5	7.5 by 5	119.0	86,000	33,000	119,000	1,000
		Manufacturing Company No. 2				
3 by 4	2.6 by 4	7.1	7,600	3,200	10,800	1,520
4 by 4	3.6 by 4	15.9	15,500	6,100	21,600	1,350
5 by 4	4.6 by 4	29.4	25,000	10,000	35,000	1,119
6 by 4	5.5 by 4	46.2	38,000	14,200	52,200	1,130
7 by 4	6.5 by 4	69.5	58,000	19,800	77,800	1,120
		Bureau of Mines Laboratory Mill				
1.575 by 3	1.575 by 3	1.54	486	890	1,376	890

[a] The mill weights are taken from catalogs. They include the pinion and accessories and the journals with their bases. It is believed that the weight of these parts is not enough to affect the deductions materially.

Both weight per unit of power and revolutions per minute are in favor of the big mills. The product of weight times speed is given in the fourth column of Table 2 (an expansion of Table 1), and the quotient of this figure divided by the net power for grinding is in the sixth column. The decreasing numerical values from top to bottom of column $\frac{C}{D}$ indicate that the net power—grinding capacity—of big mills is high with respect to the chief contributors of friction: weight and speed. Or, conversely, the friction of big mills is relatively low when compared with their net power. These numbers are only relative. Next, the meager information available will be used to obtain the "absolute" values appearing in the last column.

TABLE 2.—*Estimates of Friction Losses as Derived from Weight and Speed when Speed is* 70 *Per Cent of Theoretical Critical Speed*

Nominal Size of Mill, Ft.	R.p.m. (A)	Total Weight, (B)	$A \times B \div 1000 = C$	Net Horsepower (D)	$\frac{C}{D}$	Relation of Friction Loss to Net Power, Per Cent
			Manufacturing Company No. 1			
3 by 5	33.0	16,500	545	8.4	65	40
5 by 5	24.8	45,400	1,150	35.1	33	20
6 by 5	22.8	64,500	1,460	55.0	27	(16.4)
7 by 5	21.0	91,000	1,910	83.6	23	14
8 by 5	19.5	119,000	2,320	119.0	19	11
			Manufacturing Company No. 2			
3 by 4	33.0	10,800	356	7.1	50	32
4 by 4	28.2	21,600	610	15.9	38	24
5 by 4	24.8	35,000	870	29.4	31	19
6 by 4	22.8	52,200	1,190	46.2	26	(16.4)
7 by 4	21.0	77,800	1,630	69.5	23	14
			Bureau of Mines Laboratory Mill			
1.575 by 3	42.6	1,376	58.6	1.54	38	24

The basis of the values in the last column is the experimental evidence that the loss by friction of a 6 by 4-ft. mill is known to be 13 per cent of the motor input or 16.4 per cent of the net power into the mill. The "16.4" is in parentheses, and the other numbers in the column are in direct proportion to the $\frac{C}{D}$ values. The extremely high friction of small commercial mills is thus shown—they are too heavy and turn at too high a speed. By the same standards the laboratory mill would be expected to have a loss by friction amounting to 24 per cent of the net power. This phase of the subject will be considered later.

Possibly the friction of some of the small mills working today is more than one-fourth of the motor output. Hence, if fancy bearings are to be used to reduce friction and the deductions made thus far are correct, the small mills are first in order for improvement and, conversely, the big mills need not cause so much concern because their loss by friction is moderate.

In spite of all the whims of friction set forth by textbooks, there is evidence to justify dealing with it in the manner presented here. In an extensive test of a 6 by 4-ft. mill the loss by friction varied directly as the speed, and the speed range extended further than is met in practice. These results have been published.[2]

Similar laboratory observations are not so satisfactory. The loss by friction (watts) at three different dead weights and at various speeds is shown in Fig. 1. The curved lines show that the friction increases faster than the speed. However, although the coefficient of friction is known to change under varying conditions, the cause of this divergence is traceable to the nature of the machinery. In order to have a flexible ball-mill set-up, the mill was mounted on rollers. These could not be kept perfectly smooth, and the shell of the mill, which serves as a tire, is likewise imperfect. As a result a small amount of hammering and bouncing occurs at high speeds. These confessed imperfections must not be construed as militating against the accuracy of the general ball-mill testing because the observed, not the theoretical, losses have always been used in calculating net powers. Furthermore, as will be shown later, the losses by friction are smaller than predicted.

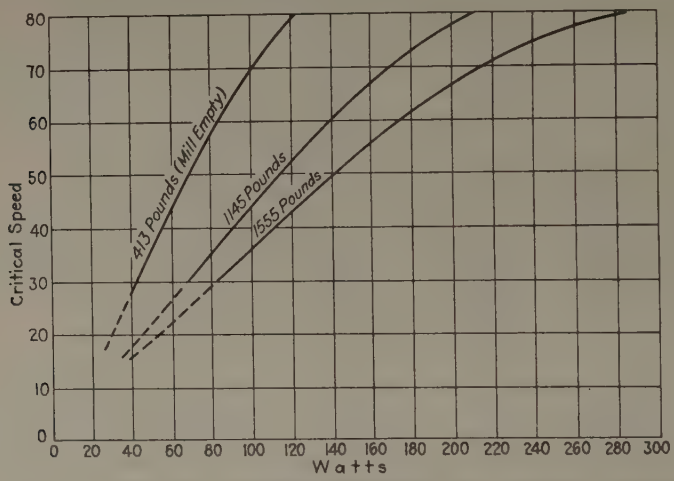

FIG. 1.—LOSS BY FRICTION AT THREE DIFFERENT DEAD WEIGHTS AND AT VARIOUS SPEEDS.

Fig. 1 shows the variation of loss by friction through various speeds when the dead weight is constant, and Fig. 2, presenting the same data differently assembled, shows how friction varies at given speeds and different dead weights. Here, again, the maximum departure from a straight line is at high speeds.

In spite of the seeming imperfection of the set-up, Fig. 2 may be used to justify the belief that friction varies as speed times weight. Pairs of these numerical values have been taken at random, with the results shown in Table 3. The ratios are seen to substantiate the theory. However, this evidence does not wholly cover the main problem to be elucidated. While it does indicate that for a given mill the friction (watts) varies approximately as speed times weight, it does not prove that for one mill the ratio of product and friction bears a definite relation to the ratio of product and friction of another mill. But, again, the designers are supposed to be consistent in their designing. If they are putting out a mill that has abnormally high friction at its regular running speed and weight they should rectify the errors.

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[2] A. M. Gow and M. Guggenheim: Dead-load Ball-mill Power Consumption. *Eng. & Min. Jnl.* (1932) **133,** 632.

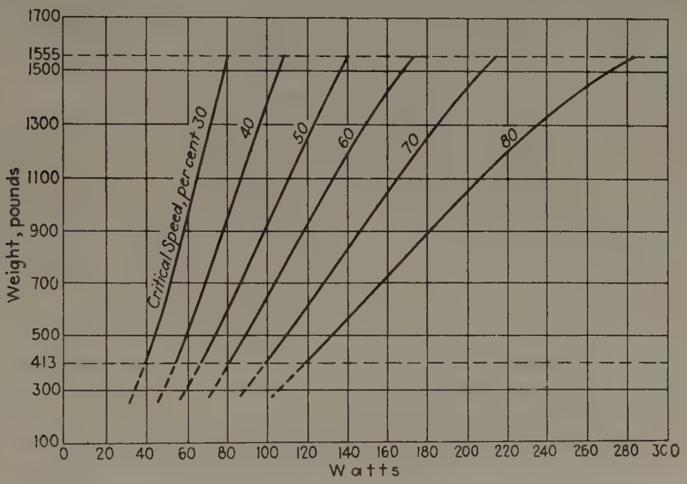

FIG. 2.—VARIATION OF FRICTION AT DIFFERENT SPEEDS AND DEAD WEIGHTS.

It will be seen that by the reasoning applied to the other mills the loss by friction would be 24 per cent. The laboratory mill does not have the ordinary babbitt bearings hypothecated for commercial mills. The bearings are the best roller bearings obtainable, and the driving is by means of a good link chain. In fact, the friction is not as high as the predicted 24 per cent. Table 4 gives more detail than is necessary for the

TABLE 3.—*Variation of Friction with Speed Times Weight*

CRITICAL SPEED, PER CENT		WEIGHT, LB.		PRODUCT (*A*)	LOSS BY FRICTION, WATTS, (*B*)	RATIO OF *B* TO *A*
50	×	1,145	=	57,000	114	1 to 500
70	×	1,555	=	109,000	215	1 to 510
50	×	1,145	=	57,200	114	1 to 500
60	×	1,555	=	93,500	173	1 to 540
60	×	1,145	=	68,700	138	1 to 497
80	×	1,555	=	124,000	282	1 to 440
50	×	1,555	=	77,500	140	1 to 555
80	×	1,145	=	91,600	207	1 to 443

TABLE 4.—*Analysis of Power*[a]

796 lb. of 2-in. balls, no ore, and approximately 75 lb. of water.

	30 Per Cent Critical Speed		40 Per Cent Critical Speed		50 Per Cent Critical Speed		60 Per Cent Critical Speed		70 Per Cent Critical Speed		80 Per Cent Critical Speed	
	Watts	Per Cent	Watts	Per Cent	Watts	Per Cent	Watts	Per Cent	Watts	Per Cent	Watts	Per Cent
Motor input	657	100.0	883	100.0	1095	100.0	1320	100.0	1558	100.0	1768	100.0
Losses:												
I^2R	107	16.3	113	12.8	120	10.9	127	9.6	134	8.6	136	7.7
Brush, 2I	20	3.0	21	2.4	21	1.9	22	1.7	23	1.5	23	1.3
Stray power	38	5.8	55	6.2	74	6.8	92	7.0	110	7.1	128	7.2
Dead load	70	10.7	93	10.5	118	10.8	143	10.8	174	11.2	216	12.2
Net power	422	64.2	601	68.1	762	69.6	936	70.9	1117	71.6	1265	71.6
Per cent dead load (loss by friction) of net power	16.6		15.5		15.5		15.3		15.6		17.1	

[a] 18-tooth sprocket, 19 by 36-in. mill; No. 74508 3-hp. Allis Chalmers motor, 220 volts, 1025 r.p.m., shunt-wound.

subject under consideration but contains valuable information to aid in comprehending the problem at large; the friction at 70 per cent of the critical speed is 15.6 per cent of the net power (Table 4). This is a little more than half of the value (24 per cent) forecast in Table 2. This deduction permits some predictions based on plant observations.

A certain 6 by 4-ft. mill is known to be wasting 11 hp. by friction. If this loss could be reduced at the ratio of 24 to 15.6 by improved mechanics about 4.0 hp. would be saved. At ¾ cents for power this would be 3.0 cents per hour. On a yearly basis of 300 days (24 hr. each) the reduced running cost would be only about $216. If the mill is small it is in most need of frictionless bearings, but if it is very large the fancy bearings are scarcely justified.

SUMMARY

This manuscript has been written in the full realization that a metallurgist is likely to get burned when he tampers with friction. A friend has already made adverse criticisms. It is hoped that the paper will bring out helpful discussions. The claim about the friction of mills is this: If a small mill and a large mill are run while centrally loaded and packed to working weights, weight times speed (r.p.m.) divided by net power should be the same for each mill. If this is not so, the one giving the smaller quotient has poor bearings.

DISCUSSION

W. L. Maxson,* Milwaukee, Wis. (written discussion).—It has been generally accepted that mills of larger diameter offer some gain in efficiency as compared with mills of smaller diameter. Because of the many interdependent variables inherent to the operation of ball mills, it is very difficult to present conclusive evidence to substantiate such claims. The author has chosen a novel method of approach to this problem and in general his findings appear to be sound. Some questions may be raised regarding the details of the methods used, but in order to verify the results, the following data have been computed.

The weight of the revolving parts, including gear, shell, heads, feeder and liners, has been divided by the projected area of the two mill bearings upon which weight revolves. The rubbing velocity between the mill journals and bearings has been determined for the 70 per cent critical speed chosen in the author's manuscript. The product of these two quantities, which we might term a friction constant for a given mill, has been divided by the ball charge for each mill. The whole numbers obtained are tabulated as follows:

Mill Size	3′ × 5′	2.210	8′ × 8′	0.360
	6′ × 5′	0.683	9′ × 8′	0.335
	8′ × 5′	0.436	10′6″ × 8′	0.241

In this tabulation the relative friction loss per unit weight of ball charge steadily decreases from the mills of smaller size to those of larger sizes.

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From the standpoint of friction losses and from an economic standpoint it is desirable that a mill should have a minimum weight for the revolving parts per unit weight of ball charge. A tabulation of these relative weights, shown as pounds of revolving parts per pound of ball charge, is as follows:

3′ × 5′	3.94	8′ × 8′	2.27
6′ × 5′	3.17	9′ × 8′	2.51
8′ × 5′	3.09	10′6″ × 8′	2.39

This tabulation likewise shows that there is less loss in dead weight per unit of ball charge for the larger mills. Such tabulations are of interest to the extent that they show that larger mills should show less loss by friction than the smaller mills but, as has already been pointed out, the percentage loss in terms of total horsepower input is relatively unimportant for the larger mills.

From a practical standpoint, the work done by a mill may to a large extent be measured by the volume of ball charge. This is definitely related to shell length and diameter. The dead-weight load for a given volume of ball charge is important and it may be exaggerated if mills are built with too short a shell for a given diameter. A rough ratio as between the weight of shell and liner as compared to heads, head liner, gear, feeder, etc., is 1 to 1.2 for relatively short mills. As the length of the shell is increased this ratio may be reversed and with exceedingly long mills the weight ratio for the heads and their relative parts may become exceedingly low.

The above relates definitely to the possible mechanical friction losses that may be incurred and that may affect a mill efficiency. It appears that these losses are not of great importance, except possibly in the mills of smaller diameter. Some very large mills have been mounted on roller bearings, using bearings up to 5 ft. in diameter, but their use has not been justified in operation.

The authors' conclusions can be generally accepted with regard to the mechanical efficiency alone, but from the standpoint of grinding efficiency it is held that with an increase in diameter of a mill, the weight of the superimposed ball mass in the "grinding toe" is increased and that in consequence there is an actual increase in the mesh tons ground per unit of power expended. This conclusion appears to be well substantiated by many comparative tests as between different sizes of mills, and it is felt that any paper dealing with the subject of relative mill efficiencies should recognize the importance of this factor.

O. H. Johnson,* Denver, Colo. (written discussion).—The note of warning sounded in the paper against mill friction should be heeded by the designer and builder of this class of equipment. It should stimulate him to check carefully into the bearing designs, sizes and construction.

The sizes of ball mills increased because it was the sound way to meet the demands for peak capacity. Their adoption lowered initial costs, saved in building expense, maintenance charges and in general made it practical to consider the milling of 2000, 5000, 10,000 and even 20,000 tons in one plant.

To illustrate the efficiency of the big mill we might recall that if Inspiration had installed what then was our standard 6-ft. machine, instead of the large mill it did adopt, it would have required more than 100 units as against 40; 13,000 sq. ft. of machine space as against 8000 ft.; 12,000 motor horsepower instead of 10,000.

From field experience, we have found the big mills to have better grinding efficiency also. In one installation, with a certain mill size, we took off the bearings and gear so as to attach them to a new mill barrel of larger diameter. We adhered to the same number of revolutions per minute, used the same ball load and the same motor, together with identical operating conditions. This change produced a considerable gain in mill output and fineness of product.

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* Manager, Marcy Mill Division, The Mine & Smelter Supply Co.

In this case the friction would be approximately the same for both sizes, but if there were any difference it would be against the bigger shell because of its extra weight. However, since the bigger shell produced more, under like conditions, it seems reasonable to assume that its advantage comes from its size, and not from any *frictional penalties* imposed upon the smallest unit.

With the demand for larger ball mills came better engineering of them. They had to be developed for heavy loads and continuous duty. The *time-out* of operation had to be kept as low as possible, because of the greater tonnages involved. This necessitated stronger materials, better bearings and finer machine work. This may explain why Mr. Coghill found the friction of big mills relatively low—they are made better.

In checking Marcy mills against the data given in this paper, we fall far below any of the B/A ratios given in Table 1. In estimating the friction losses as derived from weight and speed, we get for our 4 by 5-ft. mill a numerical value of 22 for C/D; for the 5 by 4-ft. mill a value of 22; for the 6 by 4½-ft. mill a value of 14; and for the 7 by 5-ft. mill a value of 15. These values, when reduced to absolute values, seem to be within a closer range of each other than those mentioned in the paper. Perhaps, we can therefore assume that they show Marcy-mill bearings to be fairly adequate for the various loads. I hope this may be so, because we have endeavored to be consistent with this particular detail of construction.

Mr. Coghill and his associates should be commended for the challenge to the manufacturer, to go out and cut down the evil of friction, especially in the smaller mills and in mills that may be called upon to carry greater loads than ordinarily catalogued. I am sure the urge to provide better bearings will receive attention and that betterment will come.

Some Fine-grinding Fundamentals

By A. W. Fahrenwald,* Member A.I.M.E.

Fine grinding cannot be accomplished in machines in which the component parts move in definite and restricted paths with respect to each other. Such machines are crushers. A "grinding mill" may be defined as any machine in which the crushing elements are free to make direct contact with each other but are kept from doing so by the sand to be crushed. Such a machine must possess the ability to give numerous contacts per unit of time. Upon consideration, one will see that this definition nearly includes the old Chilean and Huntington mills, the "rolls" and the stamp mill. It definitely includes all forms of ball, pebble and rod mills.

The ideal (perfect) grinding mill may be defined as one in which the crushing elements are identical in shape and substance and possess perfect freedom of motion with respect to one another. Of the mills mentioned, only the ball and pebble mills come under this definition of the ideal mill. Even the rod mill fails to get a place under this definition of the perfect mill.

Modern Fine-grinding Mill

In current practice fine grinding is performed in rotating cylinders[1] partially loaded with hard objects, the agitation of which accomplishes the crushing or grinding of the rock or ore.

The hard objects called "grinding media" nearly always are steel or some other alloy of iron. Hard rock (flint pebbles) have been and still are used to a limited extent. When steel is used as the grinding substance, the form is the sphere or "ball." The combination of rotating cylinder and steel-ball grinding media is termed "ball mill." Shapes other than the sphere have been tried. Cylindrical grinding media also have been and are now used in grinding mills. When cylindrical media are used the mill is termed "rod mill."

Mechanical Elements of Grinding Mills

The mechanical elements of a grinding mill are: (1) the rotating element, (2) the grinding media, (3) the rotating means.

Manuscript received at the office of the Institute Sept. 5, 1934.

* Professor of Metallurgy, University of Idaho, Moscow, Idaho.

[1] The rotating element may be of form other than cylindrical; for example, conical or spherical. Form of rotating element will not be discussed.

The function of the rotating element is to "agitate" the grinding media. The nature of the agitation has an important bearing on such factors as: (1) "rate" of grinding, (2) efficiency of grinding, (3) economy, and (4) metallurgical results. The nature of the media agitation as far as rotation of the mill is concerned is determined largely by: (1) the peripheral speed of rotation and (2) physical character (rough, smooth, corrugated, etc.) of the inside of the rotating element. Other factors that enter are discussed below.

The important properties of grinding media are: (1) a blend of hardness and toughness to resist wear, (2) shape of media and, (3) density.

Property No. 1 is important economically and metallurgically. High steel consumption per ton of ground product is costly and therefore will need to be held to a minimum. Iron is detrimental metallurgically in, for example, the cyanide process, where it consumes oxygen and causes inferior extractions and high reagent consumption.

Various shapes of grinding media have been experimented with, the object, of course, being to find the shape of media that gives the maximum grinding efficiency. Cubes, disks, tetrahedrons, etc., have been tried. The sphere has proved most effective.

The subject could perhaps stand further systematic study, taking into account such other important variable factors as pulp dilution and pulp load. Point contact, line contact and surface contact no doubt require different conditions of mill feed and feeding to produce maximum results.

Grinding media density is a variable that has received no systematic study. It is known, of course, that a ball mill has much greater capacity per unit of mill volume than a pebble mill. It is probably, although not necessarily, true that balls and pebbles of the same size and numbers agitated in a given cylinder at a given speed would give the same number of crushing contacts in unit time. The "flow" characteristics of steel and flint may upset this probability. If the ore piece were fully crushed in each case the capacity of the ball and pebble mill should be equal. The crushing forces, however, are not equal and with coarse feed there would be many ineffective crushing blows in the grinding media of light density. In studying grinding-media density some of the important variables to consider are: (1) feed size, and (2) pulp dilution and load. For any given feed size it would be extremely interesting, if it were possible, to vary the grinding-media density. The capacity curve thus obtained would be of much interest.

Points of Commercial Interest in Fine-grinding Mills

Some of the points of commercial interest in fine grinding are: (1) capacity or rate of grinding, (2) mechanical efficiency, (3) over-all grinding economy, (4) suitability of the ground product to subsequent metallurgical treatment.

Classification of Commercial Grinding Mills.—There are several possible bases of classification of grinding mills. Three are suggeste below:

1. Volume of pulp in the mill
    a. Overflow or high pulp-level mills
    b. Grate or low pulp-level mills
2. Speed of rotation of mill
    a. Slow speed; i.e., mills running below critical speed
    b. High speed; i.e., mills running above critical speed
3. Kind of grinding media
    a. Ball mills
    b. Pebble mills
    c. Rod mills
    d. Rock mills

The bases of classifications 1 and 2 are more fundamental than that of classification 3.

Classification of Important Variables Affecting Mill Performance.—

A. Rotating element
    1. Speed of rotation; i.e., peripheral speed
    2. Contour of mill surface in contact and supporting grinding media load
    3. Diameter of mill
    4. Length of mill or ratio of diameter to length
B. Grinding media
    1. Load
    2. Density
    3. Shape
C. Feed
    1. Size of feed
    2. Feed-grinding media ratio or feed load
    3. Feed-water ratio or pulp density
D. Circulating load, which involves ratio of reduction.

Laws of Crushing

Rittinger's[2] law of crushing as originally stated was as follows: "The energy necessary for reduction of particle size is directly proportional to the increase of surface."

It may be assumed to have been definitely proved by the researches of Geoffrey Martin and coworkers;[3] Gross and Zimmerley;[4] and Dean and Gross and Wood.[5]

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[2] P. K. von Rittinger: Lehrbuch der Aufbereitungskunde, 19. Berlin, 1867.

[3] G. Martin and others: Researches on the Theory of Fine Grinding, III. *Trans.* Ceram. Soc. (1925–26) **25**, 51, 63.

[4] J. Gross and S. R. Zimmerley: Crushing and Grinding, III.—Relation of Work Input to Surface Produced in Crushing Quartz. *Trans.* A.I.M.E. (1930) **87**, 35.

[5] E. W. Dean, J. Gross and C. E. Wood: Measurement of Crushing Resistance of Minerals by the Scleroscope. U.S. Bur. Mines *Rept. of Investigations* 3223.

Martin, using a laboratory ball mill and quartz, measured the surface of the feed and product of the mill by a dissolution method using HF as solvent. The basis of the method is Wenzel's law stated as follows: "The reaction velocity between solids and liquids is proportional to the area of contact."

The energy consumed in operating the mill was a straight-line function of the new surface produced.

Martin found that 60.9 ft-lb. of work was required to produce 1 sq. ft. of new surface.

Gross and Zimmerley, using a similar but seemingly more refined method of measuring quartz surface and a drop-weight method of crushing, also proved the existence of a straight-line relationship between energy consumed in crushing and new surface produced. For quartz these authors found that 17.56 sq. cm. (0.02692 ft-lb. per sq. in.) of quartz surface is produced for each kilogram-centimeter of work input in crushing.

R. T. Hancock,[6] who has made a careful analysis of existing data on crushing, comes to the conclusion that Kick was right and Rittinger wrong. Mr. Hancock states:

> The low estimate in which Kick's Law is today held is due singularly enough to Stadtler's advocacy of it. I do not know where the weak point in his mathematical treatment lies, but his system of ordinal numbers is all wrong. The work done is really proportional to the difference of Log D and Log d. Where D is the diameter of the initial partical and d the diameter of the final partical, or to log $\left(\frac{D}{d}\right)$.

It would seem, however, that Gaudin, Gross and Zimmerley[7] have shown experimentally and graphically that Kick's hypothesis cannot possibly hold.

Crushing Resistance of Minerals and Mineralogical Hardness

Dean, Gross and Wood,[8] using the scleroscope, have found that the energy absorbed (difference between height of drop and rebound in a

TABLE 1.—*Data on Crushing*

Mineral	Resistance to Crushing[a]	Hardness[b]
Quartz	1.00	7
Pyrite	0.788	6–6.5
Sphalerite	0.311	3.5–4
Calcite	0.231	3.0
Galena	0.192	2.5

[a] From J. Gross. [b] From standard textbooks.

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[6] Private communication Jan. 3, 1933.

[7] A. M. Gaudin, J. Gross and S. R. Zimmerley: The So-called Kick Law Applied to Fine Grinding. *Min. & Met.* (Oct., 1929) 447.

[8] Reference of footnote 5.

scleroscope) was directly proportional to the weight of material crushed per unit of work.

In Table 1, taken from the writer's ore-dressing notes, there are given for various common minerals the mineralogical hardness and resistance to crushing.

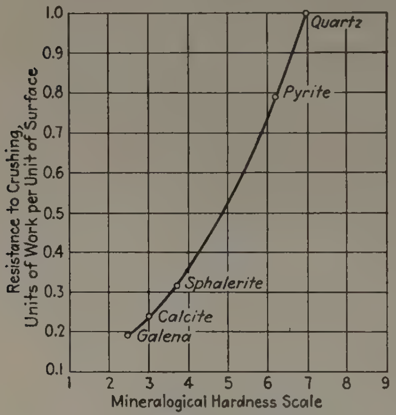

FIG. 1.—RELATION OF RESISTANCE OF MINERALS TO CRUSHING TO MINERALOGICAL HARDNESS.

When resistances to crushing, in units of work per unit of surface, are plotted as abscissas against mineralogical hardness as ordinates, the curve of Fig. 1 is obtained.

Efficiency of Crushing

The absolute efficiency of crushing may be defined as the quotient of the energy output of the crushing operation divided by the energy input. The energy input can perhaps be recorded with a satisfactory degree of accuracy. (With the drop-weight crusher, very accurately, and in a rotating mill (ball mill) with a good degree of accuracy with the wattmeter or integrating wattmeter.)

The work output, however, probably never has been accurately interpreted. Probably it is divided between two major products; namely heat and new surface. The energy equation is then:

$$E_i = C_c \times J + S \times E_s$$

Where E_i indicates energy input, in ergs,
C_c, calories of heat liberated,
J, factor converting calories to ergs,
S, new surface produced,
E_s, surface energy of substance crushed.
Letting $C_c \times J = E_h =$ work output in heat energy,
$S \times E_s = E_k =$ work output in surface energy.
Then $E_i = E_h + E_k$

A knowledge of either E_h or E_k is sufficient to compute crushing efficiency.

The work output in form of heat energy E_h probably cannot be absolutely determined experimentally. Such vitiating elements as "heat of wetting" and cooling effects due to creation of new surface come in largely as unknown factors. Fahrenwald et al,[9] by a method of determining grinding efficiencies based on thermal measurements, obtained ball-mill efficiencies ranging from 5.9 to 24.9 per cent.

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[9] A. W. Fahrenwald, G. W. Hammer, H. E. Lee and W. W. Staley: Thermal Determination of Ball-mill Efficiency. A.I.M.E. *Tech. Pub.* 416 (1931).

Work output, E_k, is the product of total surface and surface energy. Total surface probably is reasonably accurately determined by the dissolution method, at least for quartz, using HF. There are given in the literature two surface energy constants for quartz. Both values, one by Edser[10] (920 ergs per sq. cm.) and one by Martin[11] (510 ergs) are deduced on purely theoretical grounds and are of very doubtful accuracy; probably too low.

To show the wide divergence of ball-mill efficiency data as derived by the two methods—the method of determining E_h and the method of determining E_k—Table 2 is included.

TABLE 2.—*Comparison of Ball-mill Efficiency as Calculated by Thermal Means with That Resulting from Calculation Based on New Surface*

Calculations Based on	Mill Efficiency, Per Cent	
	Run A	Run B
Edser's value of 920 ergs per sq. cm.	1.19	1.03
Martin's value of 510 ergs per sq. cm.	0.624	0.574
Thermal calculations	19.7	7.4

Elasticity of Minerals and Crushing Efficiency

The elasticity of a mineral is the property that enables it to withstand temporary deformation (elastic deformation) due to external forces. The effect of elastic deformation on the mineral crystal is the same as that due to change in temperature.

Minerals that possess high elasticity when crushed should be expected to produce more heat per unit of new surface created than minerals of little or no elasticity, because of the relatively large amount of energy they are capable of absorbing within the atomic structure of the crystal.

Jeffries and Archer[12] state:

> It is known that when metals are subjected to reversed stresses, below the safe working stress, they develop a small amount of heat and cause a slight rise in the temperature of the metal. As the stress range increases, there is at first a gradual increase in the temperature of the specimen, but a stress is finally reached where the temperature rises sharply in an unmistakable manner. It has been found that the lowest stress range which produces this decided heating effect corresponds closely with the lowest stress range which produces failure in the specimen.

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[10] Edser: The Concentration of Minerals by Flotation. British Assn. for Advancement of Science. Fourth Rept. on Colloid Chemistry and Its General and Industrial Applications, 281. London, 1922.

[11] Recent Research in Science of Fine Grinding. *Jnl.* Soc. Chem. Ind. (1926) **45**, pt. 4, 160 T.

[12] Z. Jeffries and R. S. Archer: The Science of Metals, 22. New York, 1924. McGraw-Hill Book Co.

If this is true of metals, there is no reason why it is not true of non-metallic, crystalline solids, such as quartz. This is no doubt the source of most of the heat generated in ball-mill grinding and the cause of low absolute efficiency. This same idea is alluded to by Gross (reference of footnote to Table 1).

Efficiency of Commercial Ball Mills

Most ball-mill efficiency data have been obtained on small laboratory mills. Gross and Zimmerley,[13] at the Tooele plant of the International Smelting Co., with great pains obtained efficiency figures for a number of commercial ball mills. These data are given in Table 3.

TABLE 3.—*Commercial Ball-mill Efficiencies*[a]
(GROSS AND ZIMMERLEY)

	Mills		
	Primary	Secondary	Both
Power input to motor, kilowatts	260.4	213.60	474.00
Work represented in crushing	122.4	89.91	212.31
Efficiency compared to laboratory figure of 17.6 sq. cm. per kilogram-centimeter per cent	47.0	42.1	44.8

[a] These data are not absolute efficiencies.

Assuming that the efficiency of the drop-weight method of crushing represents the highest attainable crushing efficiency, there is theoretically considerable room for improvement in efficiency of ball milling. The author of this paper, however, is of the opinion that these efficiency figures are not likely to be much increased for ball mills.

Having now briefly discussed the fundamentals of crushing and having indicated the efficiencies obtained in present ball-milling operation, it is in order to take up the factors involved in ball-mill performance, which have already been classified.

VARIABLES AFFECTING BALL MILL PERFORMANCE

Speed of Rotation (Peripheral Speed)

The speed of rotation of a mill has perhaps the greatest effect on mill performance of any of the factors relating to the rotating element. The speed of a mill is given in terms of "per cent of the critical speed"—critical speed being defined as that speed of rotation at which a given ball in contact with the mill shell will centrifuge provided no slip occurs between the mill and the ball. The mathematical formula is $N = \frac{54.18}{\sqrt{S}}$

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[13] J. Gross and S. R. Zimmerley: Efficiency of Grinding Mills. U.S. Bur. Mines *Rept. of Investigations* 2952 (1929).

where N is the critical speed and S the radius of the mill in feet less the radius of the ball.

The derivation is for a single ball. Two forces act on the ball: (1) the centrifugal force required to maintain the circular motion, and (2) a component of the force of gravity due to the weight of the ball. The radial component of the force of gravity (Fig. 2) is $C = W \cos \theta$, where C equals the centripetal force, W equals the weight of the ball, and θ, the angle between the vertical axis AA' and a radius vector through the point of contact P. The centripetal force is also equal to MV^2/S where M is the mass of the ball, V its velocity ($2\pi NS$) and S the radius of rotation of the ball.

When these two forces are equal, the ball just makes a complete revolution with and in contact with the mill shell and

$$W \cos \theta = \frac{W 4\pi^2 S^2 N^2}{gS}$$

and (dividing by W)

$$\cos \theta = \frac{4\pi^2 S N^2}{g}$$

Under these conditions,

$$\cos \theta = 1$$

and

$$N = \frac{0.903}{\sqrt{S}} \text{ revolutions per second}$$

or

$$N = \frac{54.19}{\sqrt{S}} \text{ revolutions per minute.}$$

A mill operated at speeds above critical performs properly only when certain special conditions are observed and provided. This is because "ball slippage" is essential. The required conditions for ball slippage are: (1) smooth liners, (2) small ball load, (3) small feed load, and (4) pulp dilution regulation. Condition 3 requires a special discharge head, a head that will remove pulp from the mill near the shell of the mill and against centrifugal force. This special discharge head, now covered in patents pending by the author, comprises (1) what we call a pulp-slippage compartment and (2) a pulp lifter. A mill provided with this type of discharge head and operated at speeds above critical is called a "high-speed mill" as differentiated from the mill operating below critical speed or "slow-speed mill." For these reasons, present standard slow-speed mills cannot and will not function at speeds above critical. The conditions for high-speed milling were worked out in a research by Fahrenwald and Lee.[14]

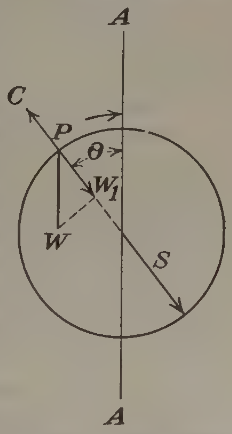

FIG. 2.—FOR DERIVATION OF FORMULA FOR SPEED OF ROTATION OF A SINGLE BALL.

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[14] A. W. Fahrenwald and H. E. Lee: Ball-mill Studies. A.I.M.E. *Tech. Pub.* 375 (1931).

High-speed mills have a much greater grinding capacity per unit volume of mill than do slow-speed mills. They consume roughly proportionately more power, grinding media and liners than slow-speed mills. Absolute, total and useful efficiencies as compared to slow-speed mills have not been well worked out, especially for mills of commercial size. Gross[15] gives some rather carefully worked out data for a mill 12 in. in diameter by 16 in. long, for conditions of batch grinding. The speeds, however, ranged only between 31 and 100 per cent critical and the data cannot, therefore, be considered for high-speed milling.

Mill speeds in present practice vary from 53 to 86 per cent of critical. Gow, Campbell and Coghill[16] in their researches, in which only speeds below critical were considered, found that a speed of 60 per cent of critical gave the maximum grinding while a speed of 50 per cent gave the most efficient grinding. The ball load, including voids, was 40 per cent of the mill volume.

Gross [17] obtained the highest total efficiency at a speed of 84 per cent of the critical speed, but at this speed his data show that the useless work (amount of over-grinding) is also at a maximum. Gross obtained the greatest useful efficiency (sand coarser than 400 mesh and finer than 200 mesh) at a mill speed of 60 per cent of the critical; a ball load of 74 per cent of the mill filled; a pulp density of 40 per cent by volume, and a small quartz charge.

Mill Surface, Diameter and Length

Mill shape has not received serious attention in the laboratory. Apparently little significance is attached to this factor as related to mill performance.

Gow, Campbell and Coghill,[18] studying laboratory ball mills operated at subcritical speeds, found that: (1) the surface tons, i.e., grinding capacity, varied as the 2.6 power of the diameter, (2) the horsepower varied as the 2.6 power of the diameter, and (3) the units of surface per unit weight varied as the 0.6 power of the diameter of the mill. Fahrenwald and Reck[19] made a similar study of small-scale ball mills operated at speeds above critical. Results were obtained as follows: (1) Mill capacity varies as the 2.65 power of mill diameter, (2) power consumption of the mill varies as the 2.65 power of the mill diameter, and (3) capacity per unit of mill volume varies as the 0.7 power of the diameter.

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[15] J. Gross: Studies of the Efficiency of Ore Crushing and Grinding. Presented at A.I.M.E. meeting in February, 1932.

[16] A. M. Gow, A. B. Campbell and W. H. Coghill: A Laboratory Investigation of Ball Milling. *Trans.* A.I.M.E. (1930) **87**, 51.

[17] Reference of footnote 15.

[18] Reference of footnote 16.

[19] A. W. Fahrenwald and Reck: Unpublished manuscript entitled "Relation of Capacity of a Small-Scale Ball Mill to Mill Length and Diameter," April, 1933.

It seems safe to say, in view of these researches, that the relation between capacity of a ball mill (small mills) and its diameter may be expressed by the formula,

$$c = KD^{2.6}$$

and power and diameter by the formula:

$$P = KD^{2.6}$$

It is probably safe to assume that these relationships also hold for commercial mills.

Experimental data from a set of runs in which the mill speed was 130 per cent of critical are shown graphically in Fig. 3. In the experiments from which these data were obtained, the ball load was 25 per cent of the mill volume, the material ground was quartz. The mills used were 6, 9, 12 and 18 in. in diameter, respectively. For each size of mill a series of three or more speeds above critical was run, to minimize chance of experimental error and to make possible comparison of the performances of the various mills at identical speeds.

Diameter constant, mill length is a factor worthy of consideration. Mill capacity is roughly but not exactly proportional to mill length. There has been no systematic study of commercial mills to determine the relation of length to general overall mill performance.

It seems obvious, since mill capacity varies as the 2.6 power of the diameter and nearly directly as the length, that the trend in mill design should be toward increasing diameter rather than length.

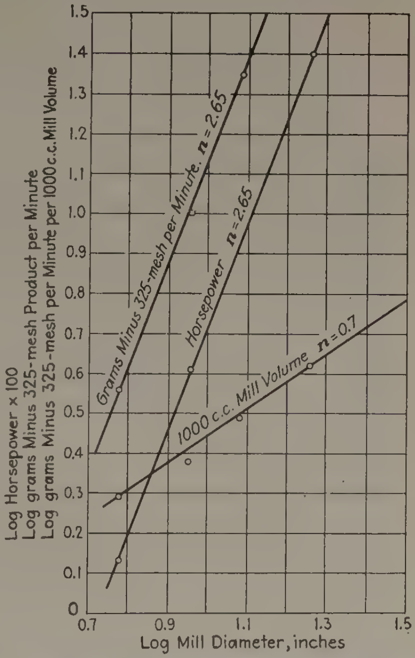

FIG. 3.—DATA SHOWING RELATION OF POWER AND CAPACITY TO MILL DIAMETER. MILL SPEED, 130 PER CENT × CRITICAL.

In a laboratory study of a 12-in. diameter mill Fahrenwald and Reck found the optimum length for maximum capacity and efficiency to be roughly two-thirds the diameter. The relationship between mill capacity and length is shown in Fig. 4.

In the experiments from which the data in Fig. 4 were taken, the mill lengths were 12, 4, 2 and 1 in. For each mill a series of runs was made in which mill speed was the variable. This was designed, of course, to locate the condition of optimum capacity for each length. These data are of interest and are given graphically in Fig. 5. The points of optimum capacity are those plotted on Fig. 4.

The data of Fig. 5 are of interest aside from the reason for their acquisition. They show: (1) that for each length of mill for a given ball load and other given feed conditions, there is a "best speed" so far as capacity is concerned; (2) that the speed for maximum mill capacity increases with mill length to what may be termed a "critical length"; (3) the best speed at the critical length is about 200 per cent of critical; and (4) that the capacity of a 12-in. mill is about twice as much at nearly 200 per cent of critical as it is at 100 per cent of critical speed and less.

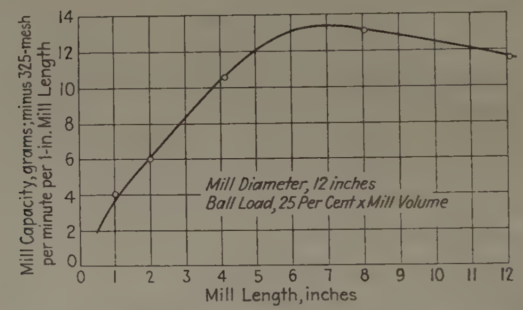

FIG. 4.—RELATION OF MILL CAPACITY TO MILL LENGTH.

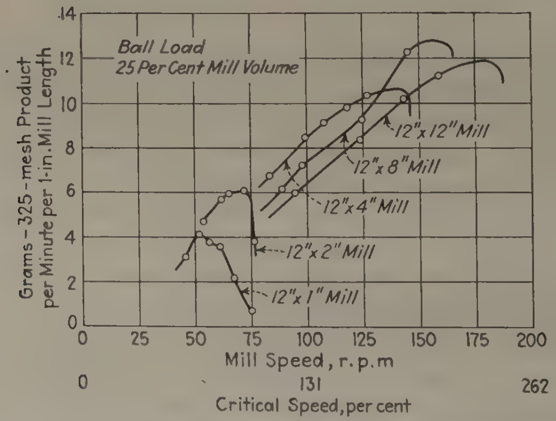

FIG. 5.—RELATION OF MILL LENGTH AND MILL SPEED TO MILL CAPACITY.

For two mills of equal volume, but of different diameters, the mill of larger diameter has the greater capacity; for example: suppose there is in operation a mill whose diameter is d_s. What is the diameter of a mill of the same capacity whose length is three-fourths that of its diameter?

Let d_s = diameter of small mill
l_s = length of small mill
c_s = capacity of small mill
d_l = diameter of large mill
l_l = length of large mill $= \dfrac{3d_l}{4}$
c_l = capacity of large mill $= c_s$

General equation expressing relation of mill capacity and mill diameter is $C = LKD^{2.6}$
where L = length of mill
D = diameter of mill

Therefore, $c_s = L_sKd_s^{2.6}$

$$c_l = L_lKd_l^{2.6}$$

and

$$L_sd_s^{2.6} = L_ld_l^{2.6}$$

and

$$d_l^{2.6} = \frac{L_sd_s^{2.6}}{L_l}$$

$$d_l = \frac{L_sd_s^{2.6}}{\frac{3}{4}d_l}$$

$$d_l^{3.6} = \tfrac{4}{3}(L_sd_s^{2.6})$$

$$d_l = \sqrt[3.6]{\frac{4L_sd_s^{2.6}}{3}}$$

GRINDING MEDIA

In the consideration of grinding media, the important factors are: (1) load in the mill, (2) density, (3) size, (4) shape and (5) resistance to wear.

Load of Grinding Media

The volume V of a mill is

$$V = \pi r^2l = 0.7854d^2l$$

where r is the radius and l the length.

The weight of a cubic foot of steel balls is 300 lb.[20] The balls occupy 62 per cent of the volume and the void space 38 per cent.

The ball load in a mill, therefore, is obtained from the following formula:

$$L = 300P(0.7854d^2l)$$

where L equals the load in pounds and P the percentage of load to be carried. The formula becomes:

$$L = 235.62Pd^2l$$

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[20] A. M. Gow: Weight of Steel Balls in a Ball Mill. *Eng. & Min. Jnl.* (1933) **134**, 203.

The power required to operate any given mill at a constant speed is variable with ball load. This statement is enlightened by reference to Fig. 6, data of which have been compiled by Fahrenwald and Lee.[21]

At speeds below critical (critical for this mill is 78 r.p.m.), the power curves have characteristic gentle slopes and the power peaks are not reached until the mill is approximately 50 per cent loaded. Increasing the ball charge simply increases the friction between the balls and the mill and causes them to assume a velocity approaching that of the mill wall.

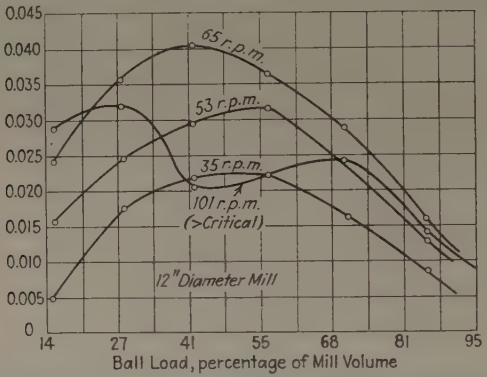

FIG. 6.—RELATION OF BALL LOAD TO HORSEPOWER CONSUMPTION AT VARIOUS MILL SPEEDS.

At high operating speeds the pheripheral velocity of the mill is so great that even small charges reach the centrifuging point and abnormal-looking curves are obtained.

Generally it may be said that the power peak corresponds to capacity in ball milling; i.e., power input to a mill and tonnage output (or surface output) run more or less parallel. Thus the power input to a mill may generally be taken as a measure of the grinding rate of that mill. This fact was clearly brought out for the first time by Fahrenwald and Lee.[22] The same thing has been more recently discovered by Campbell and Coghill.[23]

Density of Grinding Media

There has been no systematic study of grinding in a mill in which grinding-media density was the variable. This perhaps is to be explained in the fact that balls of different densities possessing satisfactory hardness properties are difficult to procure. Flint pebbles (approaching the spherical form) and steel spheres are the only materials easily available. Spheres or balls of satisfactory and of uniform hardness perhaps could be best obtained by loading hollow cast-iron balls with metal of different densities; such, for example, as lead and aluminum. Such an investigation would require numerous tests in which such factors as feed size and load and pulp density enter as variables.

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[21] Reference of footnote 14.

[22] Reference of footnote 14.

[23] A. M. Gow, M. Guggenheim, A. B. Campbell and W. H. Coghill: Ball Milling. See page 24, this volume.

Flint pebbles, used widely and in large tonnages in early cyanide practice, and still used in relatively small amounts, have the advantage of adding no fouling element to the pulp which may be harmful in subsequent metallurgical treatment.

Shape of Grinding Media

Although from time to time and at various mining districts, various shapes of grinding media have been tested, the ball is universally used. The object presumably has been to increase or improve grinding-mill performance. Cubes, disks, tetrahedra, etc., have been tried. It is a question of type of media contact and media agitation.

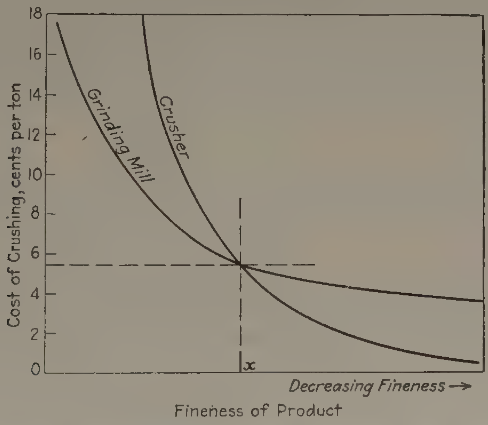

FIG. 7.—HYPOTHETICAL RELATION BETWEEN CRUSHING AND GRINDING COSTS AND FINENESS OF PRODUCT.

FEED

Size of Feed

In considering size of feed to a grinding mill the question of over-all crushing economy is paramount. Two outstanding features of the fine-grinding mills are: (1) the manner in which the grinding element is renewed and (2) the infrequent requirement of adjustment for "set." In a crusher, as defined in the first paragraph of this paper, the outstanding unfavorable features as contrasted to a grinding mill are: (1) complete replacement of crushing elements at regular intervals and (2) constant attention to maintain uniform crusher "set."

Where one type of comminution shall end and the other begin is a purely economic question. The idea is brought out hypothetically in Fig. 7. For reduction finer than x-mesh, grinding mills are more economical. If a graph of this character could serve in all cases without regard, for example, to plant tonnage and physical character of the material being reduced, a graph based on actual operating data could be developed that would be of immense practical value. This, however, cannot be done.

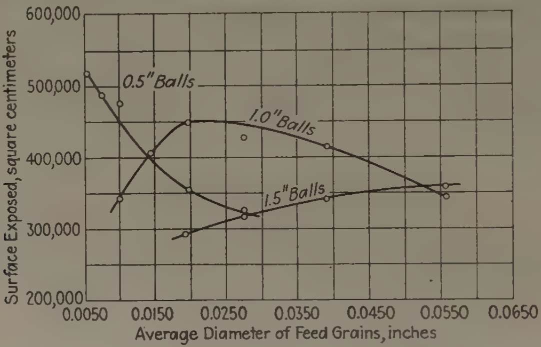

FIG. 8.—EFFECT OF GRAIN SIZE UPON MILL OUTPUT (QUARTZ).

For the moment let us disregard over-all economy and consider feed size and grinding-mill performance. Here again, it is difficult to arrive at conclusions of general usefulness. Too many variables are involved. Results of systematic experiments, however, are of interest and value, in that they provide fundamental knowledge.

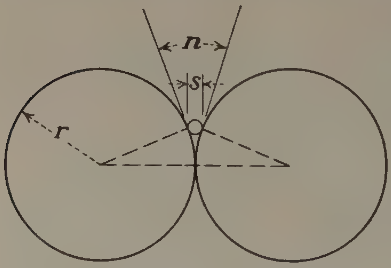

FIG. 9.—ANGLE OF NIP OF BALLS.

The graph of Fig. 8 is made up from data obtained in the laboratory[24] with a 12-in. dia. mill. For any given ball size it is plain to be seen there is an optimum feed (grain) size. Viewing the relation of ball diameter to optimum particle diameter as a function of angle or nip, the following well-known relationship holds:

$$\cos\frac{N}{2} = \frac{r + \frac{y}{2}}{r + \frac{s}{2}}$$

where r (Fig. 9) is radius of the ball, s the diameter of the particle and y the distance between the balls.

Since the balls in the mill, under conditions here considered, are very close together, and in most cases touching, the above formula becomes:

$$\cos\frac{N}{2} = \frac{r}{r + \frac{s}{2}}$$

---

[24] Fahrenwald and Lee: Reference of footnote 14.

From Fig. 8 the optimum particle size for 1-in. balls is 0.0198 in.

Thus $$\cos\frac{N}{2} = \frac{0.5}{0.5099} = 0.988$$

$$\text{angle } \frac{N}{2} = 8°50' \text{ and } N = 17°40'$$

By a similar calculation for 9⁄16-in. balls, $N = 17°$.

This value is not in agreement with the value of 45° given by Gaudin,[25] but is in close agreement with the average angle of nip of 17°26′ for rolls reported from 45 mills.[26]

The coefficient of friction between ball surface and particle is the factor probably that largely determines the angle of nip for optimum grinding. This in turn is affected by such factors as pulp dilution and fines in the pulp. That N is so nearly constant for the various sizes of balls is of considerable significance. The inference is that much of the comminution in a ball mill is effected by a "rolls" action.

Feed Load

In practice the volume of pulp in a mill is controlled by the method of discharge; i.e., (1) the "grate and lifter" type and (2) the "trunion overflow" type. There is unfortunately no provision in modern mills by which the volume of pulp can be varied to meet varying plant conditions.

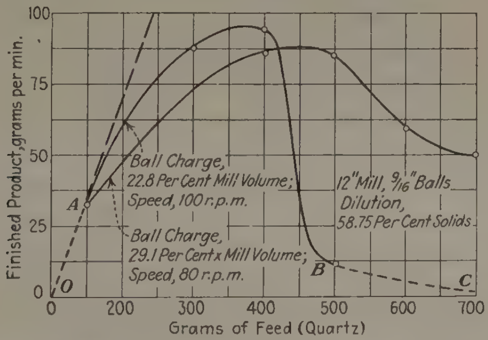

FIG. 10.—EFFECT OF FEED LOAD UPON RATE OF GRINDING.

There are no existing satisfactory data from commercial tests in which pulp load has been the variable. Laboratory studies indicate clearly that this is one of the important variable factors in ball-mill performance.

The data presented graphically in Fig. 10 bring out the importance not only of feed load but the interdependability of such factors as ball load and mill speed.

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[25] A. M. Gaudin: Investigation of Crushing Phenomena. *Trans.* A.I.M.E. (1926) **75**, 278.

[26] A. F. Taggart: Handbook of Ore Dressing, 307. New York and London, 1927. J. Wiley and Sons and Chapman and Hall.

The curves may be extended to the right and left on the basis of the assumptions: (1) that when no feed is charged to the mill, the curves must pass through the origin, and (2) that when the feed charge is increased the rate curve will approach the x axis as a limit.

In this way the curve $OABC$ is derived. From this it is evident that up to a certain limit the quantity of finished product increases approximately as a straight-line function of the quantity of feed charged. Beyond this limit the approximate straight-line relationship is modified by some function which rapidly grows in importance with the quantity of feed charged.

The deviation of the curve from the straight line is the result of several factors; namely, cushioning of the ball charge and tangential friction within the mill.

Feed-water Ratio (Pulp Density)

There are several ways in which the wetness of mill feed affects mill performance. For example: (1) wetness determines the weight or volume of solids in a mill at any given moment, i.e., the more water fed the fewer the solids in the mill, (2) wetness affects frictional forces between grinding media and particles, and (3) affects the effective density of the grinding media.

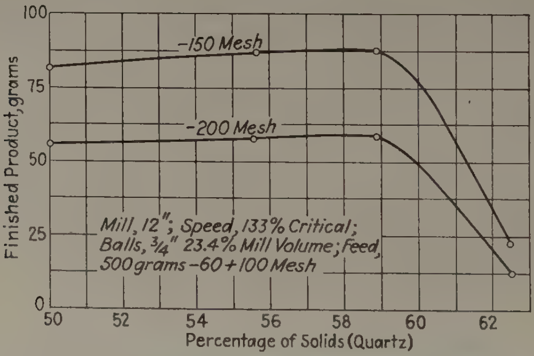

FIG. 11.—EFFECT OF CHANGES IN PULP DILUTION. Fahrenwald and Lee: reference of footnote 14, Fig. 34.

Fig. 11 contains data for a laboratory mill for two sizes of mill feed in which percentage of solids was the variable. As the percentage of solids increases, the slippage, and therefore the rolling of the balls, is somewhat decreased. This allows the balls to be carried higher within the mill. The mill output increases until the charge is raised beyond the optimum angle of break. Any further increase in the percentage of solids causes the charge to rapidly approach the centrifuging point. When this occurs, the abrupt change in the slope of the curves takes place.

Pulp density or wetness seems also to affect grinding efficiency as well as grinding rate. This is indicated in the curve of Fig. 12. These data are for a mill speed above critical and were obtained by the thermal method. However, in view of the numerous other factors involved, some of which are mentioned above, these data are not conclusive. Nevertheless, they are instructive.

Circulating Load

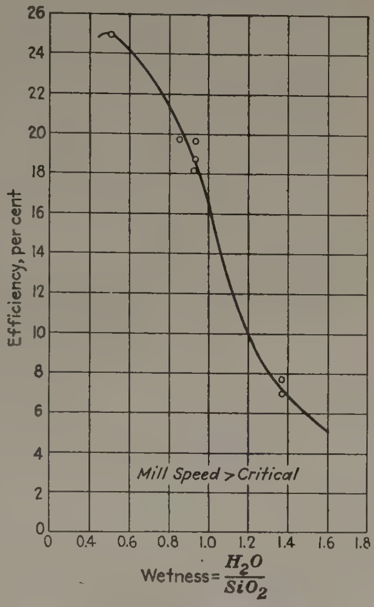

Fig. 12.—Effect of wetness upon mill efficiency.
From Fahrenwald et al.: reference of footnote 9, Fig. 1.

A ball mill that is provided with ample discharge capacity, either through means of a large discharge trunnion or pulp lifters, has a given and practically invariable pulp capacity. The time of residence of the solids in the mill is obtained from the formulas

$$R_p = \frac{C_m \times 62.5 \times D_p}{W_s + W_w}$$

Where C_m = pulp capacity of mill, cu. ft.,
D_p = pulp density,
W_s = weight solids fed, lb. per min.,
W_w = weight water fed, lb. per min.,
R_p = time of residence of solids in mill, min.

Example.—Assume a 4 by 4-ft. mill handling 2000 lb. ore per hour. The total volume of the mill is $\pi r^2 l = 50$ cu. ft. Assume the mill to be half loaded with balls. The pulp capacity of the mill is then $^{50}/_{2} \times 0.38 = 10.5$ cu. ft.

It is assumed that the specific gravity of the solids fed to the mill is 2.65 and that one part of water is added for each two parts of solids by weight. Hence

$$D_p = 66.6 \times 2.65 + 33.33 \times 1 \div 100 = 2.09$$

Using this formula: $R_p = \frac{10.5 \times 62.5 \times 2.09}{50} = \frac{1372}{50} = 27.4$ min.

Under the conditions of this illustration the pulp would be in the mill 27.4 minutes.

Now, single-pass operation never gives a product in which all the particles are reduced to finished size. The process in which the unfinished particles are hydraulically classified out of the ball-mill discharge and automatically returned to the feed end of the mill, along with the original feed, is termed "closed-circuit" grinding and the returned portion is called the "circulating load." The circulating load builds up in weight equal to several times that of the original feed.

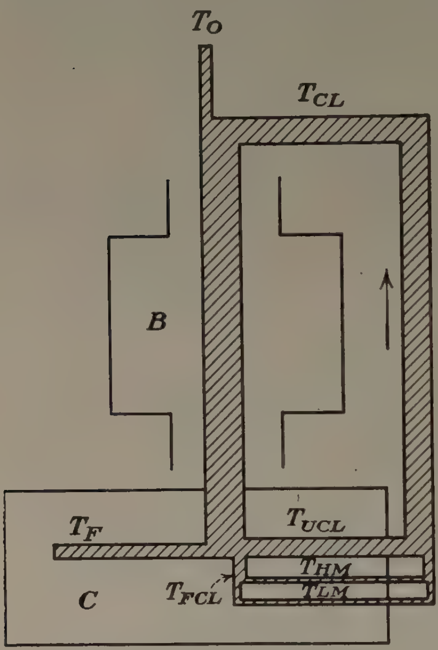

FIG. 13.—DIAGRAMMATIC PRESENTATION OF CLOSED-CIRCUIT GRINDING.

B, ball mill; *C*, classifier. Heavy black lines indicate flow of solids in system, width of lines indicating magnitude or weight of solids. Solids are designated as follows:

T_o, weight (tonnage) of original feed
T_f, tonnage of finished feed
T_{cl}, tonnage of circulating load
T_{ucl}, tonnage of unfinished circulating load
T_{fcl}, tonnage of finished circulating load

Now $T_o = T_f$
and $T_{cl} = T_{ucl} + T_{fcl}$
also $T_{fcl} = T_{hm} + T_{lm}$
where T_{hm} = tonnage of heavy mineral
and T_{lm} = tonnage of light mineral.

Unfortunately the circulating load always contains a certain percentage of "finished product," which should be removed by the classifier operating in the circuit. Because the mechanical classifier operates on the laws of free and hindered settling, the classifier is incapable[27] of separating small heavy mineral from larger lighter ones. It is here that the unit flotation cell in the circuit functions to aid not only the classifier but the grinding mill as well.

The subject is worthy of further analysis. In Fig. 13 is shown diagrammatically the closed-circuit grinding system.

Classifier inefficiency is responsible for the part T_{lm} in the circuit. This tonnage is ever present in the system and probably cannot be removed by classification. It can be minimized by efficient classification.

Tonnage T_{hm} in the circulating load theoretically and practically cannot be removed by classification methods, and when classifier efficiency is being considered this material and tonnage should not be charged against classifier efficiency. It can be removed, at least in part, by flotation, with advantage[28] both metallurgically and mechanically.

There is the good possibility, too, that even the part T_{lm} of the circulating load may be advantageously removed by methods of nonmetallic flotation.

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[27] A. W. Fahrenwald: Classifier Efficiency; an Experimental Study. *Trans.* A.I.M.E. (1930) **87,** 82.

[28] A. W. Fahrenwald: Coarse Sand Flotation. U.S. Bur. Mines *Rept. of Investigations* 2921 (1929).

Time of Residence of Pulp in Mill

Circulating load, for one thing, involves, as has already been said, time of residence of pulp in the mill. The effect of this variable on mill capacity is clearly brought out in Fig. 14.

In this test the mill was stopped at the end of 3 min., 6 min., 9 min., etc., the charge removed and sieve analyzed and returned to the mill. The percentage of the various sizes of products in the mill at the ends of the various periods of grind was recorded and plotted in Fig. 14. It may be observed from this figure that the amount of quartz ground is almost a linear function of time until approximately 55 per cent of the charge is finished. That the rate of grinding, however, is not exactly linear with time is shown by the curves of Fig. 15.

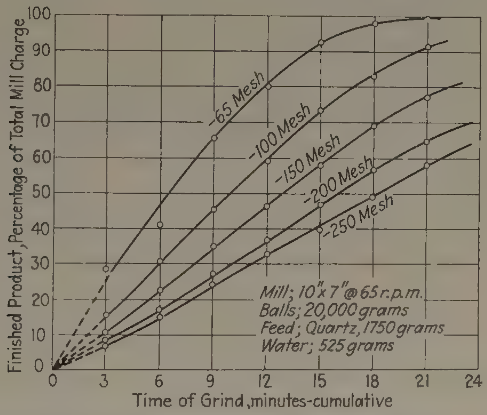

FIG. 14.—RELATION OF TIME OF RESIDENCE OF CHARGE IN MILL TO RATE OF PRODUCTION OF FINISHED PRODUCT.
From A. W. Fahrenwald: U. S. Bur. Mines R. I. 2989.

Here rate of grinding in grams per minute is plotted against time increments of grinding. For minus 150, 200 and 250-mesh products there is an increasing rate of grinding during approximately four 3-min. periods, or, in other words, during the first 12 min. of grinding. For the 100-mesh product the rate curve is nearly constant for 12 min. and the 65-mesh product curve falls off rapidly at the start.

On the basis of Rittinger's theory, it was reasoned that the rate of grinding probably should bear a direct relation to the total surface of unfinished grains in the mill which it had to work upon at any particular instance. Using the surface constants of quartz as given by Gross and Zimmerley,[29] the average total surface of unfinished sand in the mill during each 3-min. grinding period was calculated. These data are plotted in Fig. 16. By comparison of Figs. 15 and 16 it will be noticed that the rate of grinding curves and surface curves shows a close parallelism. Taking, for example, the 200-mesh product, the maximum rise of the curves occurs at from 12 to 15 min. of grinding. In view of these data a generality or hypothesis may be stated as follows:

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[29] J. Gross and S. R. Zimmerley: Crushing and Grinding, II. *Trans.* A.I.M.E. (1930) **87**, 27.

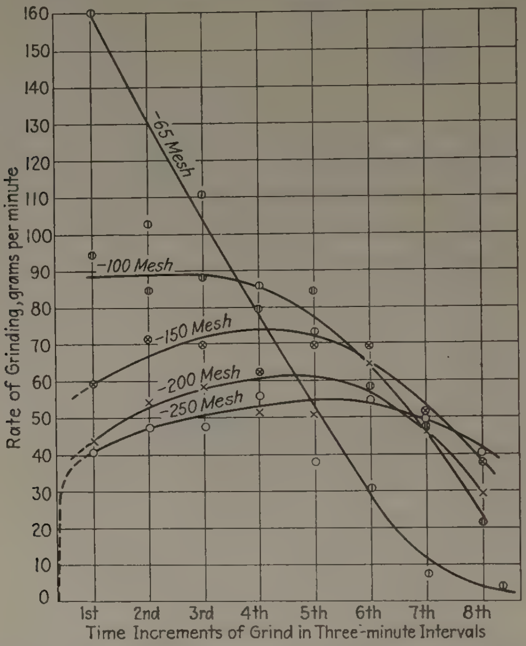

FIG. 15.—RELATION OF RATE OF GRINDING TO TIME OF RESIDENCE OF CHARGE IN MILL.

Under the condition of maximum mill load (weight of sand to be ground) the capacity of a given mill operated under given conditions is directly proportional to the number of sand grains in the mill that are greater in diameter than the dimension of the mesh through which everything is desired to be ground.

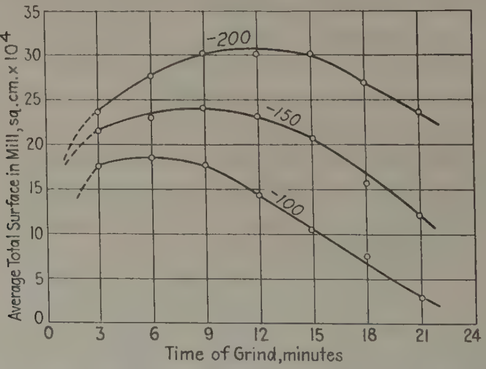

FIG. 16.—RATE OF CHANGE OF TOTAL SURFACE IN MILL WITH TIME OF GRIND.

For "number of particles" may be substituted "total surface of particles." Obviously the total surface of particles or total number of particles would be greatest: (1) when the unfinished grains in the mill are just larger than the finished mesh opening, and (2) when the percentage of finished grains in the mill was a maximum. More data bearing on this matter are compiled in Fig. 18.

In each case the initial minute of grind results in a relatively low grinding rate, because the quantity of unfinished surface in the mill is relatively low. The magnitude of the unfinished surface increases with time of grind until a maximum is reached when the number of particles just larger than the limiting mesh reaches a maximum.

In the grinding tests described above, it is obvious that the initial minutes of grinding were being consumed in preparing the feed in the mill to give maximum grinding rate. While the mill was accomplishing this it was slowly having the unground feed in the mill reduced and replaced at the same rate with finished solids. Thus, although the size of feed in the mill slowly became more favorable the load (by weight) of unfinished feed rapidly decreased.

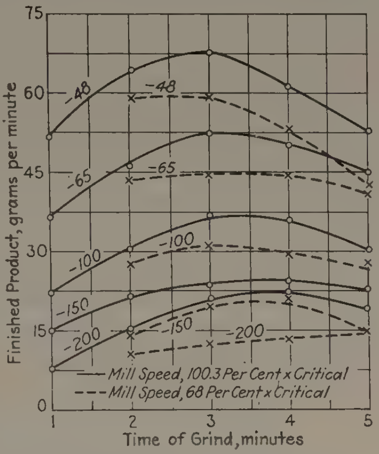

FIG. 17.—RELATION OF TIME OF GRINDING TO GRINDING RATE.

A study of this condition of affairs led to development of a laboratory closed-circuit batch grinding technique designed to obviate the above conditions and at the same time to simulate practical closed-circuit grinding.

Closed-circuit Batch Grinding

The flow sheet of the process[30] is shown in Figure 18. The desired approximate circulating load employed in any test is determined by the assigned value of y. The smaller y (in minutes), the larger the circulating load. Also for any assigned value of y the circulating load is a dependent variable with size of finished product. The finer the finished product, of course, the larger the circulating load.

A complete closed-circuit batch grind, including several cycles, is required for each size of finished product upon which data are desired. Also, sieve analyses of the return sand and the finished sand of each cycle are necessary to enable the experimenter to ascertain when that stage of the operation (number of cycles) had been reached in which

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[30] A. W. Fahrenwald: Batch Closed Circuit Grinding. U.S. Bur. Mines *Rept. of Investigations* 2990 (1930).

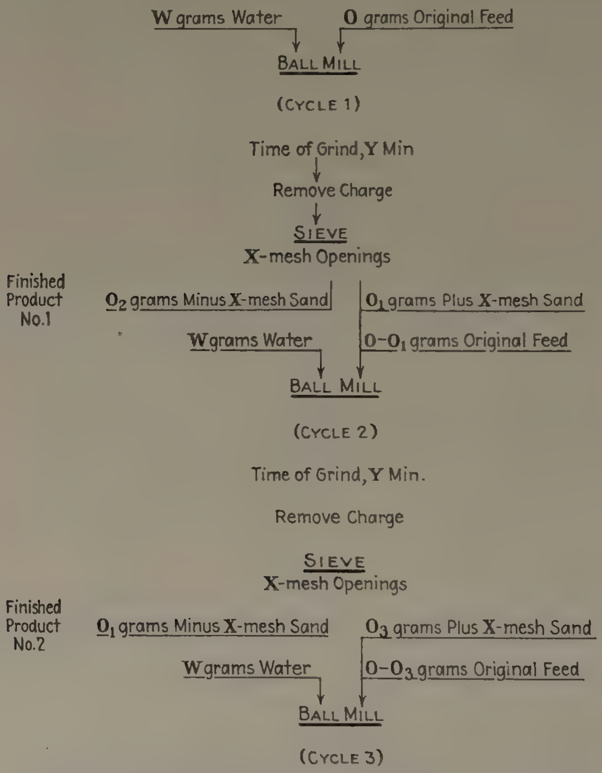

FIG. 18.—FLOW SHEET OF CLOSED-CIRCUIT BATCH GRIND. Fahrenwald and Lee: reference of footnote 14, Fig. 28.

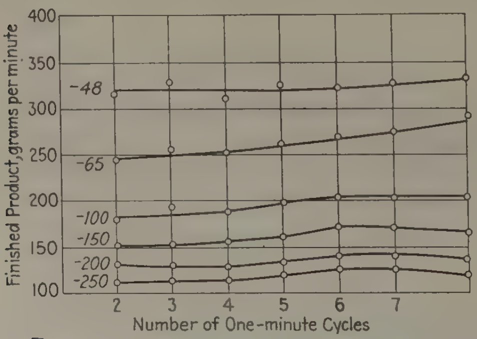

FIG. 19.—RELATION OF MILL OUTPUT TO NUMBER OF CYCLES.

the return sand had a nearly consistent sieve analysis. The assumption was made that when this stage of the closed-circuit grind had been approximately reached, the system had attained its maximum production rate and that the operation then was comparable to the practical closed-circuit system of grinding.

The data of this study are shown graphically in Figs. 19, 20, 21 and 22. Figs. 19, 20 and 21 are for tests in which the cycle y was 1 min., 2 min. and 3 min., respectively. From these data it may be observed: (1) that there is an increase in rate of grinding during the first several cycles and (2) that the rate of grinding approaches a constant which, although theoretically never reached, is practically reached at the sixth or seventh cycle.

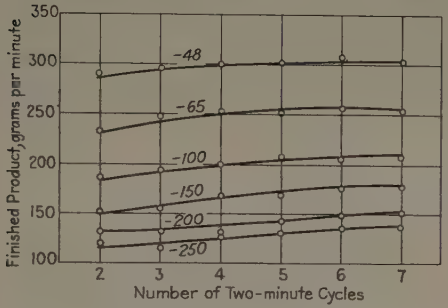

FIG. 20.—RELATION OF MILL OUTPUT TO NUMBER OF CYCLES.

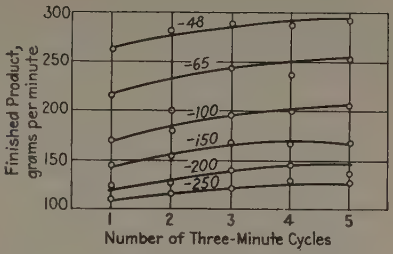

FIG. 21.—RELATION OF MILL OUTPUT TO NUMBER OF CYCLES.

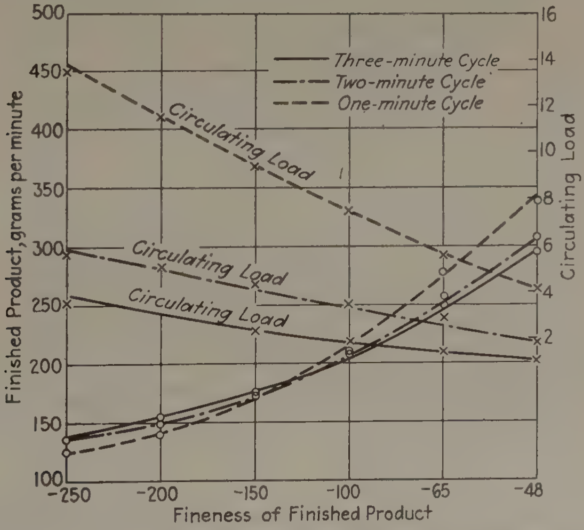

FIG. 22.—RELATIONSHIP OF CIRCULATING LOAD, SIZE OF FINISHED PRODUCT AND GRINDING CAPACITY.

In Fig. 22, rate of grinding is plotted vertically against fineness of finished product, for the 1, 2 and 3-min. cycles. These data show that for relatively coarse grinding (+100 mesh) the short (1 min.) cycle (large circulating load) gives highest mill capacity and that for relatively fine grinding (approximately −100 mesh) the 2 and 3-min. cycles give larger capacity than the 1-min. cycle. The "capacity-fineness of grinding" curves nearly coincide at the 100 to 150-mesh product.

On this same figure are plotted circulating loads against fineness of finished product for each of the cycles.

To show the trend of the change in the sieve analysis of the return sand as the cyclic operation continues, the sieve analysis data for the run in which the cycle was 1 min. and the finished product 65 mesh is given in Table 4.

It is of interest and importance that there is a gradual decrease in ‘mean mesh." There is no easy way of determining the total surface, or surface of unit weight of the return sand, but it increases as the mean mesh diminishes; hence, since the weight of return sand and original feed to the mill remains substantially constant, it is concluded that the explanation of the increase in mill capacity with each additional cycle of grind for the several cycles is to be attributed to the increase in surface of the feed in accordance with the theory stated above.

TABLE 4.—*Screen Analyses of Returned Sand between Cycles*[a]
y Equal 1 Min. Finished Product, 100 Mesh

Cycle No.	Percentages with Following Screens								Hypothetical Mean Mesh[b]
	+14	+20	+28	+35	+48	+65	+100	−100	
1	15.5	25.0	25.0	18.0	16.5				32.39
2	8.5	18.0	23.5	19.0	13.0	9.0	8.0	1.0	41.42
3	7.2	14.0	23.1	18.7	16.0	10.3	9.7	1.0	41.00
4	5.5	11.5	20.0	18.8	18.0	12.0	12.5	1.0	43.99
5	5.0	10.0	18.0	19.5	19.0	14.0	14.0	1.5	47.79
6	5.0	9.5	15.5	17.5	19.5	15.5	16.0	1.5	48.49
7	4.5	8.0	11.0	17.0	20.0	18.0	20.0	1.5	52.56
8	3.5	7.7	11.7	15.4	18.9	17.7	23.6	1.5	59.80

[a] Data from U.S. Bur. Mines *Rept. of Investigations* 2990.

[b] Minus 100 mesh not included in calculation.

Another point of interest in this study was the sieve analysis of the various finished products as affected by the magnitude of the circulating load. These data (not given here) in a general way showed that the higher the circulating load, within the limits of this research, the less finely ground was the finished product.

These tests illustrate how the rapid removal of finished sand and return of unfinished sand to the mill automatically builds up a feed of "best sieve analysis" for the mill to work upon. These data thus bring out the advantage of, and the reason for, closed-circuit grinding.

Relation of Fineness of Grinding to Mill Capacity[31]

It has been demonstrated conclusively that the energy expended in grinding is proportional to the new surface produced; i.e., for a given ball mill operated at given conditions the surface produced in two units of time is twice that produced in one unit of time. Also, it probably is true that the energy expended in minus 200-mesh grinding is to the energy expended in minus 100-mesh grinding as the surfaces produced, although this has not been shown experimentally. Whether this is true or not is of no great importance to the operator, and in any case he does not have at his command a suitable method of surface determination. What is of importance, however, is the relative capacity of a ball mill when grinding through various sieve meshes.

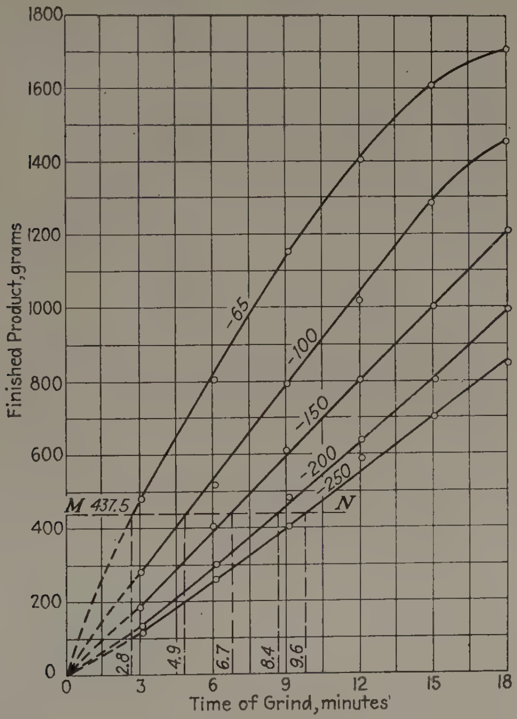

FIG. 23.—RELATION OF GRINDING RATE AND FINENESS OF FINISHED PRODUCT.

Laboratory studies with determinations of this kind in view were undertaken, and the data given here, and their analyses, may be of interest to operators.

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[31] A. W. Fahrenwald: Relation of Fineness of Grinding to Mill Capacity. Presented at A.I.M.E. meeting, New York, Feb. 15, 1932.

The data for this study are taken from a paper by the author,[32] revised and presented graphically in Fig. 23.

From these data, it is easy graphically to determine the time of grinding necessary to produce a given constant amount of finished size through any mesh. Let us grind until, say, 25 per cent (437.50 grams) of the charge in the mill has been finished. Draw a horizontal line *MN* through the ordinate on the 25 per cent point and then drop vertical lines through the points of intersection of this line with the various mesh curves. The points of intersection of these vertical lines with the abscissas give the time of grinding required respectively to produce a given quantity of the products of various sizes. The data taken in this manner from Fig. 23 are shown in Table 5.

TABLE 5.—*Relation of Output to Fineness of Finished Product*
Grinding White Quartz

Size of Finished Product		Time of Grinding to Produce 437.5 Grams of Quartz	Grams Produced per Minute
Mesh	Microns		
− 65	208	2.8	156
−100	147	4.9	88
−150	104	6.7	64
−200	74	8.4	51
−250	61	9.6	45

Plotting grams per minute on the ordinate vs. size (in microns) of finished product, i.e., the size of the sieve opening through which the product passed, on the abscissa, the curve Fig. 24 is obtained. If the grinding were continued until the finished product approached molecular size, the curve would pass substantially through the (0.0) coordinates and would of necessity approximate the shape indicated by the dotted extension *AB* of the solid curve *BC*.

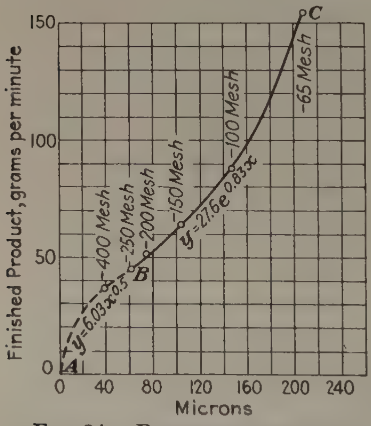

FIG. 24.—RELATION BETWEEN FINENESS OF GRINDING AND MILL CAPACITY.

Evidently the size-capacity curve follows one law down to a fineness of approximately 250 to 400 mesh and another law below this fineness. The *BC* part of the curve suggests the exponential or compound interest law and the *AB* part of the curve the power law. To test this the data are plotted logarithmically in Figs. 25 and 26. In Fig. 25 the log of grams per minute is plotted against the size of finished product in microns. The points are substantially on a straight line, the general equation of which is

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[32] A. W. Fahrenwald: Grinding and Classification, I. Batch Grinding. U.S. Bur. Mines *Rept. of Investigations* 2989 (1930).

$$Y = Mx + B.$$
$$M = \text{slope of line} = 0.36$$
$$B = Y \text{ intercept} = 1.44.$$

Thus

$$Y = 0.36x + 1.44.$$

The equation of the curve Fig. 24 then is

$$\log Y = 0.36x + \log 27.6$$

or

$$Y = 27.6(10^{.36x})$$

Converting to Napierian base e,

$$Y = 27.6e^{0.83x}$$

Thus the relation of capacity to fineness of finished product between 65 and 250 mesh appears, in the case of quartz and for the particular grinding conditions employed, to follow the compound interest law, the mathematical expression of which is $Y = ae^{bx}$.

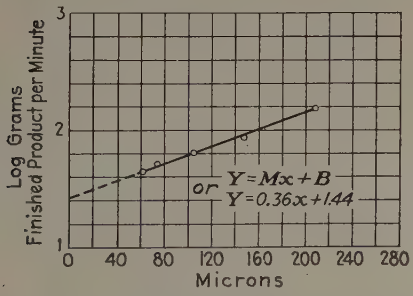

FIG. 25.

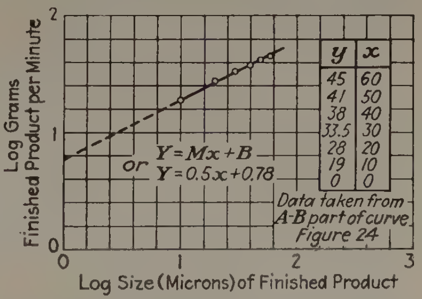

FIG. 26.

FIG. 25.—LOGARITHMIC RELATION OF FINENESS OF GRINDING TO MILL CAPACITY.

FIG. 26.—LOGARITHMIC RELATION OF FINENESS OF GRINDING TO MILL CAPACITY OF −200-MESH SIZE.

The data on Fig. 26 are obtained graphically from the dotted hypothetical part AB of curve Fig. 2. The curve of Fig. 26 is the result of plotting the logarithm of grams finished product per minute vs. logarithm of finished size in microns. The points fall substantially on a straight line, which is proof of the power law, the equation of which is $Y = 6.03x^{0.50}$. Of course, the position of the AB portion of the curve is purely a guess; however, since it must pass through the (0.0) coordinates, the shape suggested is entirely plausible.

Summary of Investigation on Work in Crushing*

By John Gross,† Member A.I.M.E.

The study of the energy used in crushing operations has advanced from a period of perplexing confusion to one of greater clarity. Only within the last few years has any true conception of the work in crushing been possible, owing to the chaotic state of theoretical knowledge. For some 20 years a controversy existed between those advocating the Rittinger law and those advocating the Kick law. The Kick law now appears to be definitely out of the picture, and crushing efficiency may be based confidently on the Rittinger theory. There also seems to be a generally crystallized conception as to the meaning and nature of subsieve material.

There is needed a simple, direct method for determining surface on a crushed ore product, and also a simple and accepted method for size determination on minus 400-mesh material.

The Crushing Law

The Rittinger(1)‡ theory of crushing was advanced in 1867 and the Kick(3) theory in 1885. While the Rittinger theory might have been accepted without much question and actually had been accepted by many investigators, a serious doubt as to its validity was raised by Stadler(10) who upon purely theoretical grounds proclaimed the Kick "law" as the true law of crushing.

According to Rittinger the energy required in successive steps in reduction increases geometrically, while according to Kick the energy increases arithmetically. These "laws" therefore are at variance, and the establishment of the true law was recognized as of first importance.

Many simple experiments have been made to demonstrate the application of the Rittinger theory to crushing, and it would seem that its simplicity should favor its acceptance. Experimental evidence, however, was not convincing, as no reliable data could be obtained on the finer material from a crushing or grinding operation. Gates,(13) Bell,(19) and others had realized that the work expended on subsieve sizes represented a very large part of the total work input.

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* Manuscript received at the office of the Institute August 31, 1934. Published by permission of the Director, U.S. Bureau of Mines.

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‡ Numbers in parentheses refer to the bibliography at the end of the paper.

The literature is ample evidence that surface figures or average size of the minus 200-mesh portion of a crushed product was entirely ignored or was at best a more or less intelligent guess. As the subsieve portion of a crushed product usually accounts for the greatest part of the work expended, calculations based on accurate figures for the coarser portion and highly doubtful ones for the finer material must be of questionable value.

A great deal of excellent work has been done in connection with the work in crushing. The crushing-surface diagram of Gates(13) is especially worthy of mention; possibly, if built upon sieve and elutriation sizes, this diagram might, by extrapolation, yield a surface figure of acceptable accuracy.

An excellent survey of research on crushing has been published by Shaw,(76) but as much of this work is now of historic interest only the appended bibliography will suffice to cover this period of investigation.

It is necessary sometimes to wander in darkness for a certain period until the time is ripe for action, and when this time arrives it often happens that light is discovered independently by several investigators. The need of a method for surface measurement finally led to a simultaneous solution of the problem. Martin,(46) in England, and the U.S. Bureau of Mines at Salt Lake City,(42) working independently and unknown to each other, evolved a dissolution method for the measurement of the surface of quartz. It is rather strange that quartz should have been used in both of these methods and that the methods were very similar. Both Martin and the Bureau of Mines, in quite a different manner, proved that the amount of surface produced in crushing quartz was directly proportional to the work expended thereon. There appears to be little doubt that the Rittinger theory is the true law of crushing.

In the experiments made by the Bureau of Mines(68) the quartz was crushed in a drop-weight apparatus in which the work going to crushing was carefully determined, and the surface produced in the crushing operation was determined by the dissolution method. The results are given in Table 1.

As the amount of surface produced per unit of work input is a constant, within the allowable error by the Chauvenet criterion, the results confirm the Rittinger theory as applying to a crushing operation.

Determination of Work in Crushing

With the law of crushing firmly established, the two requisites in the determination of the work in crushing are: (1) An accurate figure of the work input and (2) an accurate determination of the amount of surface produced.

TABLE 1.—*Relation between Work Input and Surface Produced in Crushing Quartz*

Grams Crushed	Work in Crushing, Kg-cm. per Gram		Surface, Sq. Cm. per Gram			Surface Produced, Sq. Cm. per Kg-cm.
	Total	Net	Crushed Product	Before Crushing	Due to Crushing	
72	3.42	3.02	129.7	72.3	57.4	19.0
64	10.25	9.21	227.1	72.3	154.8	16.8
20	10.25	9.37	249.6	72.3	177.3	18.9
80	15.37	10.64	393.4	200.4	193.0	18.1
40	15.37	10.64	400.0	200.4	199.6	18.8
72	15.37	10.82	406.5	200.4	206.1	19.0
48	20.50	14.77	328.1	72.3	255.8	17.3
32	20.50	14.79	349.2	97.4	251.8	17.0
296	20.50	15.04	340.3	97.4	242.9	16.2
56	20.50	16.12	343.1	72.3	270.8	16.8
108	34.17	19.85	690.2	369.8	320.4	16.1
96	41.00	25.43	518.2	72.3	445.9	17.5
16	58.70	46.45	878.4	34.2	844.2	18.2
		206.15			3620.0	17.56

Determination of Work Input.—For research purposes the work input should be that actually expended on the material being crushed irrespective of all transmission and friction losses. Haultain(34) employed a very ingenious differential integrator applied to rolls, the work input being recorded automatically. Later this device was used on a ball mill by the U.S. Bureau of Mines(90) at Salt Lake City. Coghill,(83) Martin,(50) and Fahrenwald(91) depended on electrical measurements of power.

For comparative purposes in mill operations work input to the motor may be used quite justifiably. Obviously, the results so obtained are not scientifically comparable with those obtained from a different machine.

Determination of Surface Produced.—Surface determinations have been based upon methods which may be classified as follows: (1) Sieving; (2) sieving and elutriation or sedimentation; (3) microscopic count; (4) light transmission and other methods; (5) size distribution; (6) direct measurement. All of these except the last are methods for the determination of size and therefore are only indirectly applicable to surface determinations. The irregularity of the surface of any crushed material makes it necessary to apply a correction factor which will vary with different substances.

Sieving.—In all of the earlier work sieving was the sole recourse in arriving at a surface figure. As the minus 200-mesh material was either ignored or given an assumed value, no material progress was made or could have been made in the study of the work in crushing. Sieving, however, is an important adjunct in this study. The care and conditions under which sieve analyses should be made were studied under the auspices of the Milling Committee of the A.I.M.E. and the U.S. Bureau of Mines, which resulted in the acceptance by the American Standards Association of a tentative standard method.(101)

Coghill(95) uses a method based entirely on sieve analyses for determining the work in crushing. Surface figures are made reciprocally proportional to the relative size of the sieve openings, and the mill is credited for grinding only to a limiting size. The relationship of horsepower-hours to surface produced is taken as the efficiency. This method has simplicity to recommend it for comparative results in mill operation, but it does not give a true measure of work in crushing.

Sieving and Elutriation or Sedimentation.—The further sizing by elutriation or sedimentation of the subsieve portion of a crushed product may be considered an extension of the sieve series. Sizing by elutriation should be confined to material finer than 400-mesh. The contention that this sieve is not accurate may be strictly true, but its order of accuracy is far higher than that of elutriation. When elutriation or sedimentation follows sieve sizing, a much closer approximation to surface figures may be obtained than by sieving alone. Elutriation has been used to a great extent in the study of grinding, and a variety of methods and designs has been devised.

Some 20 per cent of the references given in the appended bibliography discuss elutriation, usually accompanied by the description of a particular kind of elutriator. The Safety in Mines Research Board in England has also done a great deal of work on elutriation.

All of these efforts to simplify elutriation or to increase its accuracy suggest that a standard is greatly needed. Such a standard, both as to method and apparatus, might be selected by a competent commission that should keep in mind the basic requirements of simplicity of design and ease of operation. If made available to the industry, such an elutriator, with standard rules of operation to continue the sieve series, would eventually lead to its use almost as universally as sieves.

As a sizing device the elutriator leaves much to be desired. The density and shape of the particle affect the results irrespective of proper control of temperature and eddy currents. Elutriation, however, is the only mechanical means whereby reasonably large amounts of material may be separated into sizes finer than the finest sieve, and it should therefore come into more general use.

A method has been devised by the U.S. Bureau of Mines(78) for the determination of the surface of a ground ore by sieve and elutriation sizing. This method is based on a comparison with the actually determined surface of quartz for the various sizes, a correction being applied for density and irregularity of the various minerals that make up the ore. While this method is somewhat tedious, requiring not only sieving and

TABLE 2.—*Surface Figures for Various Minerals*

SQUARE CENTIMETERS PER GRAM

Size	¾	4/6	6/8	8/10	10/14	14/20	20/28	28/35	35/48	48/65	65/100	100/150	150/200	E_1	E_2	E_3	E_4
Quartz (sp. gr., 2.65):																	
Average diameter, microns	5,690	4,013	2,845	2,007	1,410	1,001	711	503	356	252	178	126	89	38	20	7	1.5
Theoretical surface	3.98	5.64	7.96	11.3	16.1	22.6	31.8	45.0	63.6	89.8	127	180	254	596	1,130	3,240	15,100
Actual surface	34.2	43.9	52.2	59.7	72.3	89.3	107.4	139.9	176.5	236.8	314.7	425.7	547.2	1,150	2,100	5,400	24,000
Ratio, actual to theoretical	8.59	7.78	6.56	5.28	4.49	3.95	3.38	3.11	2.77	2.64	2.48	2.37	2.15	1.93	1.86	1.66	1.59
Galena (sp. gr., 7.43):																	
Average diameter, microns	5,690	4,013	2,845	2,007	1,410	1,001	711	503	356	252	178	126	89	16	8.2	2.9	0.62
Theoretical surface	1.42	2.01	2.84	4.02	5.73	8.07	11.4	16.1	22.7	32.0	45.4	64.1	90.7	505	985	2,780	13,000
Actual surface	5.8	7.6	9.9	12.5	16.4	21.1	26.9	35.7	47.2	63.0	85.8	115.4	155.1	732.3	1,380	3,750	16,900
Ratio, actual to theoretical	4.10	3.80	3.47	3.10	2.87	2.62	2.36	2.22	2.08	1.97	1.89	1.80	1.71	1.45	1.40	1.35	1.30
Sphalerite (sp. gr., 4.00):																	
Average diameter, microns	5,690	4,013	2,845	2,007	1,410	1,001	711	503	356	252	178	126	89	25	13.4	4.7	1.01
Theoretical surface	2.64	3.74	5.27	7.47	10.6	15.0	21.1	29.8	42.1	59.5	84.3	119	169	600	1,120	3,190	14,900
Actual surface	13.1	16.8	21.6	27.3	35.0	45.0	56.8	74.5	97.7	130.9	176.2	236.8	314.3	972	1,750	4,820	21,500
Ratio, actual to theoretical	4.95	4.50	4.09	3.65	3.30	3.00	2.69	2.50	2.32	2.20	2.09	1.99	1.86	1.62	1.56	1.51	1.44
Pyrite (sp. gr., 5.00):																	
Average diameter, microns	5,690	4,013	2,845	2,007	1,410	1,001	711	503	356	252	178	126	89	23	12.2	4.3	0.92
Theoretical surface	2.11	2.99	4.22	5.98	8.51	12.0	16.9	23.9	33.7	47.6	67.4	95.2	135	522	985	2,790	13,000
Actual surface	12.1	15.6	19.7	24.8	31.4	39.8	50.7	65.7	85.6	113.8	153.0	204.7	274.1	913.5	1,650	4,460	19,900
Ratio, actual to theoretical	5.75	5.22	4.66	4.14	3.69	3.32	3.00	2.75	2.54	2.39	2.27	2.15	2.03	1.75	1.68	1.60	1.53
Calcite (sp. gr., 2.71):																	
Average diameter, microns	5,690	4,013	2,845	2,007	1,410	1,001	711	503	356	252	178	126	89	30	16.0	5.6	1.20
Theoretical surface	3.89	5.52	7.78	11.0	15.7	22.1	31.1	44.0	62.2	87.9	124	176	249	738	1,380	3,950	18,500
Actual surface	15.9	21.0	27.0	34.1	45.1	57.9	73.4	97.7	129.4	173.2	234.4	316.8	425.8	1,140	2,010	5,450	24,400
Ratio, actual to theoretical	4.10	3.80	3.47	3.10	2.87	2.62	2.36	2.22	2.08	1.97	1.89	1.80	1.71	1.54	1.46	1.38	1.32

elutriation but also determination of the main mineral constituents of the sized products, it does give approximately accurate figures.

The method gives efficiency figures for a crushing or grinding operation and portrays the part that overgrinding or useless crushing plays in efficiency.

Table 2 gives the surface figures that have been worked out for several minerals. These figures were obtained before sieves finer than 200 mesh were used, and the elutriation figures therefore are on minus 200-mesh size. The elutriation products were obtained as follows:

E_1. Material not rising in an upward current of 1.92 mm. per second.
E_2. Material not rising in an upward current of 0.48 mm. per second.
E_3. Material not rising in an upward current of 0.12 mm. per second.
E_4. Material rising in an upward current of 0.12 mm. per second.

This table is given tentatively as a possible aid in the surface determination of an ore. It needs extension to other minerals and should be carried out to 400 mesh. The figures given, however, may be utilized with the assurance that surface figures will be obtained much more readily than by any other method.

Microscopic Count.—Determination of particle size by microscopic count has received considerable attention. Improvements have been made in the preparation of the sample for examination, in the use of quartz lenses, and of the ultraviolet ray for the measurement of finer particles. Even with the exercise of great care and with the ability gained from experience microscopic measurement can only cover a field limited by the visibility of the particles. At the limit of visibility the accuracy of measurement is necessarily low. Material finer than this limit is missed entirely, and, while the amount of the material may be small, the enormous specific surface represented by it makes it important.

With proper dispersion of the sample microscopic measurements may be made, but as the particles tend to orient themselves with their longest dimension at right angles to the line of vision the particle size as determined will be too large and a correction must be applied.

The microscope should be considered an auxiliary in the measurement of particle size rather than a primary method. It is of great importance in research, but whether it has any large field for surface determination in the ore-dressing field is questionable.

Light Transmission and Other Methods.—A number of methods have been proposed for the measurement of particle size and surface based upon the amount of light transmitted through a suspension of the material. Wagner(110) has utilized this method in his Turbidimeter for measuring of specific surface of cement. The use of this appliance in determining particle size and surface of an ore involves certain corrections for the different minerals. Gooskov(72) proposed moving pictures as a method for analyzing falling particles in sedimentation, and Aborn(80) used the

X-ray to measure particle size in silica. Fahrenwald(92) has attempted to measure work in crushing by the heat produced in ball-mill grinding. As the heat accompanying the production of new surface must be very small, it would seem that any thermal measurements in ball-milling would measure mainly heat of friction, which would have no relation to the work accomplished.

Size Distribution.—Determination of size distribution and the law of such distribution have received a great deal of attention. If the size distribution of any particular crushed product is known over an adequate field, an estimate becomes possible for the finest material if a law of particle-size distribution exists. This method promises to become a help in determining the work in crushing. There are, however, two requisites for its adoption: (1) an accepted law of size distribution and (2) an accepted method of elutriation.

1. A number of investigators have been and are interested in establishing the law of size distribution. The work of Martin,(37) Gaudin,(54) Andreasen,(70) Work,(72) Hatch and Choate,(73) Aborn,(80) Roller,(94) Rosin and Rammler,(115) and Weinig(124) has been of inestimable benefit in preparing the field for a general acceptance of the law.

2. In most cases sieve sizing does not give enough data to establish the size distribution, particularly on a ball-mill product. Elutriation in some form or other must be resorted to, and the accuracy of determining the size distribution depends entirely on the accuracy of the elutriation.

Direct Measurement.—Direct measurement of surface is based on some specific property of the surface, such as heat of wetting, adsorption, or rate of solubility. The properties of heat of wetting and of adsorption cannot well be utilized for surface measurement, as the reactions therefrom do not give sufficiently large measurable quantities. The rate of solubility for the measurement of surface appears to have been first used by Wolff(27) in determining the surface of glass powder.

The dissolution method devised by the U.S. Bureau of mines and by Martin depends on the rate of solubility of quartz in hydrofluoric acid. The method is therefore restricted to the measurement of the surface of quartz. It is based on Wenzell's law, which states that, all other things being equal, the rate at which solution takes place is a direct function of the surface undergoing dissolution.

As the dissolution method is based on the solubility of the solid and as this solubility depends on the exposed surface, it follows that the surface measurement by this method is independent of shape or size of particle, which makes it an accurate method for surface measurement.

It is highly desirable that a dissolution method be developed for other minerals, but no suitable solvent for other minerals is yet available, although Kreige(52) has published a dissolution method for limestone.

The application of the dissolution method to a heterogeneous material, such as an ore, appears at this time to be hopeless.

SIGNIFICANCE OF THE RITTINGER THEORY

From rather crude observations, Rittinger evidently assumed that all the energy of crushing went into new surface. Distribution of this energy is explained quite differently now, as the best estimates indicate that the energy used in producing new surface is very small.

In a crushing operation the crystal lattice must be elastically deformed. When the deformation reaches the point of rupture new surface is produced and the strain is released in a zone proportional to this surface, and appears as heat. When the deformation falls short of rupture no new surface is produced and the strain is released as energy to be returned to the crushing appliance.

The energy used in breaking a substance either in compression or tension is the product of the breaking load and the distance through which it acts. The actual energy expended in producing new surface obviously follows the Rittinger law, and as crushing follows this law the breaking load is directly proportional to the cross-section that is related to the area of new surface produced by a proportionality factor characteristic of the substance. This proportionality factor of the Rittinger theory is determined by the distance through which the force acts in breaking and the nature of the fracture surface. This is a constant for any given material. It is designated as the Rittinger constant, and is an index of grindability. For practical purposes it is desirable to know this constant for various substances and to know how it may vary with speed and method of breaking.

Koster,(128) from a mathematical analysis of fracture, finds that fracture is not determined by a definite load but by a definite rate of deformation. Even the most brittle substances will adjust themselves to a slowly applied load, and fracture will not occur until the rate of load application or deformation has reached a certain amount. The Rittinger constant will depend on the rate of load application, and it may be predicted that the energy required to produce fracture will be less with rapidly applied loads.

Koster has given formulas for calculating the Rittinger constant, but the numerical data required in making quantitative use of the formulas are not available. Possibly, however, the Rittinger constant, that is, a grindability or crushing resistance figure, may be determined by the use of the scleroscope.(127) It has been found that the energy absorbed, or difference between height of drop and rebound, is directly proportional to the weight of material crushed per unit of work for quartz, pyrite, calcite, sphalerite and galena. This method is so simple and apparently logical that it offers possibilities of standardization to determine grindability.

Finally, the Rittinger constant apparently represents 100 per cent efficiency in crushing, and a further approach toward this limit must depend on the utilization of principles not found in present crushing or grinding appliances. Such an increase in efficiency may be accomplished by instantaneous crushing, in which the elastic deformation of the material would be virtually eliminated owing to the suddenness of the blow; by differential crushing, where the ore mineral would be disintegrated rather than the gangue; and by elimination of useless crushing in the production of undesirable fines.

All of these possibilities would seem to be demonstrated by explosive shattering. The sudden explosive force would tend to eliminate the elastic deformation. Ore minerals usually are more porous than gangue minerals, and as the adhesive tension between unlike minerals is less than the cohesive tension in a uniform solid, selective crushing may be obtained. It has generally been found that less undesirable fine material is produced in explosive crushing than in ball milling.

Conclusion

The advance made in the study of the work in crushing has been encouraging, and with the elimination of the Kick "law" future investigative work will be on a firm basis.

Many investigators have devoted an enormous amount of time and effort to advancing knowledge on crushing, and the few pages of summary given in this paper can touch only the more salient features, many data and much information being necessarily omitted.

A general survey of the crushing problem indicates that the most pressing needs to which investigative efforts should be directed are:

1. A method for surface determination that is fairly accurate, simple and applicable to an ore.
2. A standardized apparatus and method of elutriation or sedimentation that can be introduced into regular practice.
3. Establishment of the law of particle-size distribution in a crushed product.
4. Investigation of means to accomplish comminution other than those now employed.

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Grindability of Various Ores

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Grinding is one of the major problems in present-day milling practice, and in many cases, it represents one of the main items of expense. It becomes necessary from time to time, to compare grinding practice in one plant with that in another plant, and in making such comparisons, it would be very desirable were there available accepted standards of grinding resistance for the materials involved. Such a set of grinding constants would also be of considerable value in planning new grinding installations.

There have been some tests published, notably those of Lennox,[1] which have been generally cited as a basis for such comparisons. Most of the tests made have been based upon open-circuit grinding, in which a weighed sample of the ore was crushed to pass a certain mesh, and was then ground under certain conditions, either wet or dry. Grinding was continued until a certain desired fineness was attained, and the comparisons based on the grinding time, or the samples were all ground for a certain specified length of time and comparison based upon the screen analysis of the products.

Such methods have been acceptable where the ore was to be ground in open circuit. However, in modern milling practice, many grinding problems involve closed circuits wherein the oversize returns to the mill and a large circulating load builds up. A method of testing that does not take the circulating load into consideration might yield results quite different from those to be expected in practice, because the minerals in the ore that offer maximum resistance to grinding will accumulate in the circulating load up to an equilibrium value. Thus the nature of the ore charge in the mill at any instant may differ greatly from that of the new feed, with a consequent error in the rate of grinding determined by such a method.

For example, let it be assumed that an ore to be tested consists of equal parts of difficult grinding mineral and easy grinding mineral. This is to be compared with an ore used as a standard, and consisting of minerals which are all of about the same grindability. If a comparison were made between the two, based on a short period of grinding in a ball mill, the ore under test might well appear to be extremely easy to grind, since the easy grinding mineral would be rapidly reduced. However, if the test were continued for a long period the large amount of fines from the easy grinding fraction would retard the rate of reduction of the difficult grinding fraction, and the grindability obtained might be entirely erroneous.

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[1] L. W. Lennox: Grinding Resistance of Various Ores. *Trans.* A. I. M. E. (1919) **61**, 237.

The method described below, which makes it possible to measure the closed-circuit grinding characteristics of an ore, was developed in the mining laboratory of the Allis-Chalmers Manufacturing Co. It is intended to measure the relative grindabilities, only at certain specified meshes, and under conditions analogous to those obtaining in ball, tube and pebble mills. The relative ease of coarse crushing, and of grinding under conditions where impact is the chief factor, such as in a stamp battery, may be very different from the relative grindabilities as determined here.

Open-circuit grinding tests are made only when the material is to be ground in open circuit.

Method of Determining Grindability

A sample of the ore weighing about 40 lb. is crushed through rolls, screened through 6 mesh, and the oversize returned to the rolls until the sample is all minus 6 mesh.

This weight of sample is sufficient for several tests if grindabilities are desired at several different meshes, or if a check test is desirable. It has been our experience that a minus 6-mesh particle of feed is small enough to be broken and nipped by the balls used; and crushing the feed finer than necessary is objectionable, since it brings the average size of the feed closer to that of the product.

A screen analysis of a split sample of the minus 6-mesh material is made, using the sieves in the Tyler standard screen scale. A sample is placed in a 1000-c.c. graduate (57-mm. dia.) and compacted by shaking to yield a volume of 700 c.c., and containing a minimum amount of voids. The apparent specific gravity and weight in pounds per cubic foot are computed. The "unit volume" consists of 700 c.c., since that is the volume of sample always present in the mill during a closed-circuit grinding test.

We have chosen to use a standard volume rather than a standard weight in the laboratory mill, because in the parallel grinding of two ores of different specific gravities in a full-sized mill the volumes of the ore charges at any one time will be more nearly equal to each other than will their weights.

The largest volume that can be conveniently screened at one time in three half-height Tyler sieves in a Ro-Tap is 700 c.c.

The ball mill used is a cylindrical mill, 12 by 12 in. inside, which operates at 65.8 r.p.m. It contains a charge of 285 iron balls weighing 20,125 grams. The balls range from 1-½ to ¾ in. in diameter. The weight and the total number of balls are kept constant and corrected for loss by replacing the smallest ball present with a larger one, thus maintaining an approximately constant grinding surface.

The volume of the mill is 1358 cu. in., or 22,200 c.c.; the volume of the ball charge without voids is 2810 c.c.; and the volume of the ball voids is 1920 c.c., making the volume of the ball charge with voids 4730 c.c. These measurements were made by displacement of water. The ball voids are 40.6 per cent of the volume of the ball charge with voids. The volume of the ball charge with voids is 21.3 per cent of the total volume of the mill. The volume of the ore charge (700 c.c.) is 36.4 per cent of the volume of the ball voids. These constants were chosen for convenience in operation rather than to obtain the maximum mill efficiency, since the object of the tests is to obtain an accurate comparison of different ores, rather than to arrive at absolute values.

A unit volume of the dry minus 6-mesh sample is placed in the mill, and the mill is run for the number of revolutions (measured by a revolution counter) considered best from previous experience on similar ores. The mill contents are then removed and the balls screened out. Assuming that the grindability test is to be made at 100 mesh, the mill discharge is screened through three 100-mesh sieves placed in the Ro-Tap for 10 min., and the minus 100-mesh portion is weighed and the weight tabulated as the mill product. All tests are made with a 250 per cent circulating load; thus the minus 100-mesh portion should weigh 1/3.5 of the unit volume charged into the mill. This ratio is obtained by varying the number of revolutions for successive grinding periods.

Sufficient amounts of fresh sample are added to the plus 100-mesh portion of the mill discharge to replace the minus 100-mesh material screened out, thus bringing the total weight of the charge back to the original weight, which is again charged into the ball mill. The plus 100-mesh portion of the mill discharge is called the circulating load. The weight of the minus 100-mesh material in the original feed, as secured from the screen analysis, is subtracted from the weight of the minus 100-mesh material in the mill discharge, and the remainder, which is called the "net product," is divided by the number of revolutions to obtain the "net production" in "grams per revolution." From this figure the number of revolutions required for the succeeding period can be anticipated.

These cycles are continued until the net production in grams per revolution reaches a constant numerical value, and a condition of equilibrium is established. Screen analyses of the circulating load and of the mill product are made following the last period.

At equilibrium the average grain size of the circulating load and of the mill product are relatively constant. This equilibrium value for the net production in grams per revolution is called the "grindability" of the material, and is obtained by averaging the last few production figures after equilibrium has been established. From 7 to 15 grinding periods usually are required to establish equilibrium. After equilibrium has been reached the net production in grams per revolution for succeeding periods will usually check within about 3 per cent, and the average gives a fairly accurate figure. It is to be noted that in some cases the net production from successive periods increases, while in other cases it decreases as the grinding test progresses.

A typical data sheet showing a grindability test on Homestake gold ore at minus 48-mesh follows. This is an ore of about average grindability.

Unit volume (700 c.c.) = 1335 grams
Apparent specific gravity = 1.91, equivalent to 119 lb. per cu. ft.
True specific gravity = 3.12, voids = 38.8 per cent
Minus 48-mesh in feed = 21.9 per cent

The charge for the first period consisted of 274 grams of new feed and 1061 grams from the circulating load and the plus 48-mesh portion of the mill product from the previous test at minus 28 mesh.

Period	Revolutions, 65.8 R.p.m.	−48-Mesh in Product, Grams (382)	−48-Mesh in Feed, Grams (21.9 %)	−48-Mesh Net Product, Grams	Net Product, Grams per Revolution
1	110	330	60	270	2.46
2	126	402	72	330	2.62
3	112	392	88	304	2.71
4	109	385	86	299	2.74
5	109	393	84	309	2.83
6	105	383	86	297	2.83
7	105	385	84	301	2.87
8	104	395	84	311	2.99
9	99	378	86	292	2.95
10	101	377	83	294	2.91
Averages..........	101.3	383.3	84.3	299	2.95

Grindability—average of last three periods = 2.95 grams per revolution
Circulating load—average of last three periods = 248 per cent
Required to produce unit volume of minus 48-mesh = 453 revolutions
Screening time = 5 minutes

The following equation has been derived from theoretical consideration, and expresses the relationship existing between the grindability and the other terms involved after equilibrium has been reached where:

X = grindability in grams per revolution
R = number of revolutions per period

F = grams of ore in mill (weight of unit volume)
U = percentage of undersize in feed divided by 100.

Then:

$$X = \frac{F}{3.5R(1 + U + U^2 + U^3 + U^4 + \cdots)}$$

The variation of the results of any test from this relationship is a measure of the experimental error involved. It is usually within 1 per cent.

Grindability tests have been made at 28, 48, 100 and 200 mesh on several ores on which operating data such as power consumption, screen analyses of feed and finished product, mill capacity, etc., are available. These tests are used as standards, and after a test has been made on any ore its grinding characteristics may be computed by comparison of its grindability with that of one or more similar ores used as standards and ground to the same mesh. Such tests have already been made on many different materials, chiefly ore samples, and operating data obtained from commercial installations have served to verify the results obtained from these tests.

Fig. 1 is a graph of the results of various tests, and examination shows a number of interesting features, which may be enumerated as follows:

1. The difference between the grindabilities of various materials continually decreases with finer grinding. This is to be expected when it is considered that with increasing fineness of grinding there is an increasing amount of breaking of individual mineral grains, while at the coarser meshes much of the work done in grinding consists in unlocking the grains from one another.

2. Pure quartz is the easiest to grind of any of the materials of low specific gravity tested at minus 28 mesh, and one of the hardest at minus 200 mesh. The quartz is practically homogeneous.

TABLE 1.—*Grindability and Specific Gravity*

Ore	Grindability at 200 Mesh	Specific Gravity	Grind. at 200 Mesh, Per Cent of Quartz	Specific Gravity, Per Cent of Quartz
Quartz	0.878	2.65	100.0	100.0
Portland	1.035	2.69	118.0	101.5
Miami	1.139	2.69	129.8	101.5
Homestake	1.260	3.12	143.6	117.6
Bingham	1.854	4.65	211.1	175.4

3. The grindability, in general, is higher for the ores of higher specific gravity. Table 1 shows that the grindability at minus 200 mesh increases in the same order as the specific gravity; but that the differences are

considerably greater than can be accounted for by the difference in specific gravity, and these increases are still more evident with coarser grinding.

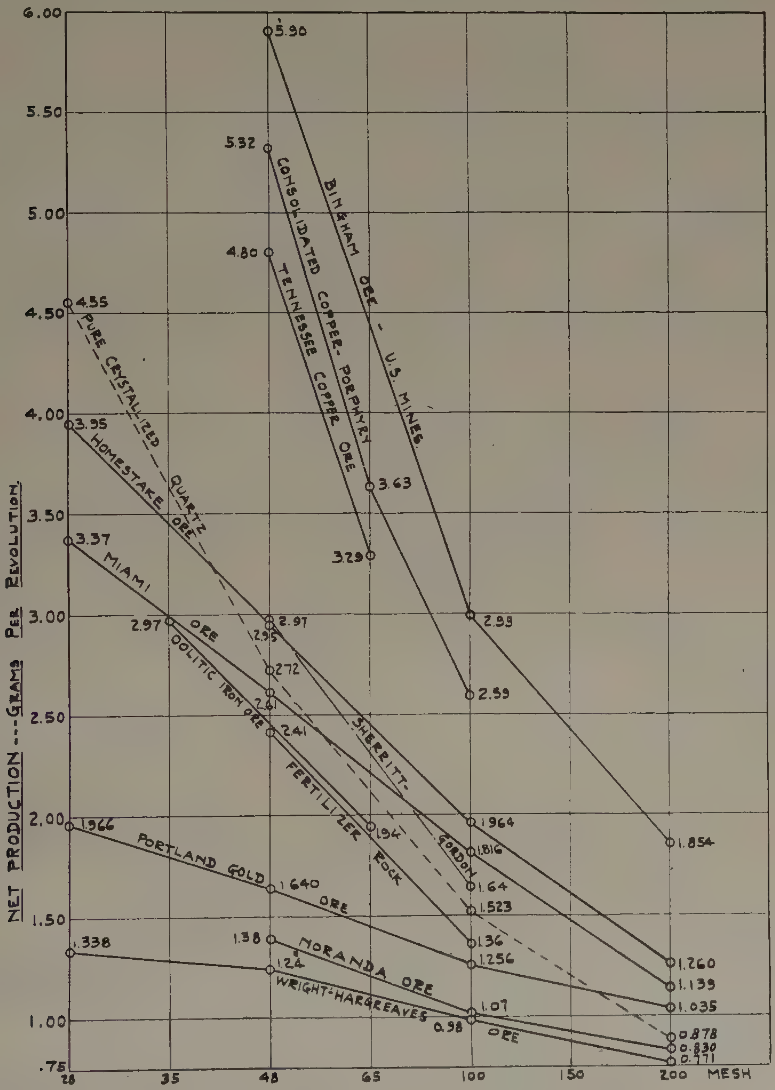

FIG. 1.—GRINDABILITY TESTS AT VARIOUS MESHES.

4. The grindability of any ore at different meshes tends to follow a line that is almost straight for the harder ores, such as Portland, but tends to be concave upward for the ores that are easier to grind.

Fig. 2 is a graph showing the relative ease of grinding equal volumes of the ores plotted in Fig. 1. It is to be noted that the grindabilities of different ores on a volume basis are closer together than when compared on a weight basis, and this should be borne in mind when comparing commercial installations that are grinding heavy ores.

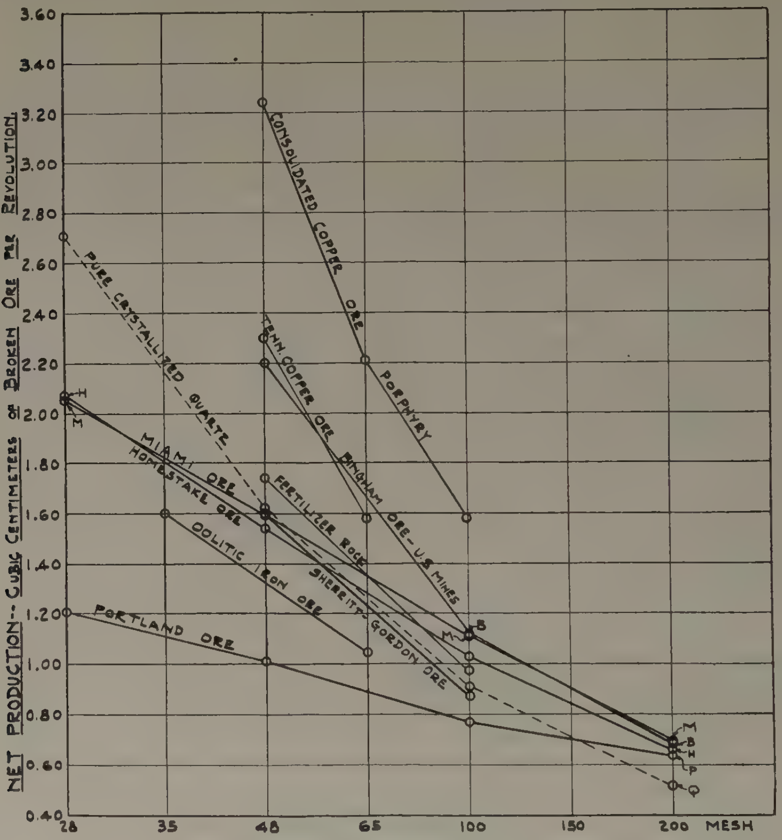

FIG. 2.—RELATIVE EASE OF GRINDING ON A VOLUME BASIS.

METHOD OF EVALUATING SCREEN ANALYSES

It is evident that considerable variation may exist in the average grain diameters of different samples all ground to pass the same screen. A method for computing the average grain diameter of a sample from its screen analysis is given below.

The procedure often used, of assigning a definite diameter to the minus 200-mesh material, was rejected, since it is known that different samples that are all minus 200 mesh may have widely different average grain diameters. Instead, the "median diameter method" was developed,[2] in which the diameter of the median grain is calculated. Each screen in the Tyler standard screen scale is assigned a number, and the number (n) of the hypothetical screen that would divide the sample into two portions of equal weight is computed from the cumulative screen analysis. From this number the diameter of the median grain is calculated from the formula:

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[2] F. C. Bond: Calculating Average Particle Size from Screen Analysis, *Eng. & Min. Jnl.* (1931) **132.**

$$\text{Median diameter} = \frac{8052}{(1.414)^n}$$

where the numerator is the average grain diameter retained on a number 0, or 3-mesh, screen, and the denominator is the square root of 2 raised to the power n.

Where 50 per cent or more of the sample is retained on 200 mesh the value of n is calculated as follows:

Let n_1 represent the number of the finest sieve retaining less than 50 per cent cumulative of the sample, and n_2 represent the number of the coarsest sieve retaining more than 50 per cent, or $n_2 = n_1 + 1$. Then

$$n = n_1 + \frac{50 - \text{per cent cum. on } n_1}{\text{per cent cum. on } n_2 - \text{per cent cum. on } n_1}.$$

When the log of 1.414, or 0.1505, is multiplied by n, and the product subtracted from the log of 8052, or 3.906, the difference is the log of the

TABLE 2.—*Screen Number and Grain Diameter*
TYLER STANDARD SCREEN SCALE

Screen No.	Mesh	Opening, Microns	Average Diameter of Grains Retained on Screen, Microns
−4	Inch 1	26,670	32,180
−3	¾	18,850	22,760
−2	½	13,330	16,090
−1	⅜	9,423	11,376
0	Mesh 3	6,680	8,052
+1	4	4,699	5,694
2	6	3,327	4,026
3	8	2,362	2,847
4	10	1,666	2,013
5	14	1,178	1,423
6	20	833	1,006.5
7	28	589	711.7
8	35	417	503.2
9	48	295	355.8
10	65	208	251.6
11	100	147	177.9
12	150	104	125.8
13	200	74	88.96
14	270	52	62.91
15	400	37	44.48

median diameter in microns, or thousandths of a millimeter. This entire calculation can be made with three settings of a slide rule.

TABLE 3.—*Screens Not in Tyler Standard Screen Scale*

Mesh	Opening, Microns	Screen Number (n)
10	1900	3.621
12	1520	4 265
14	1300	4.716
16	1130	5.121
18	980	5.531
20	864	5.895
30	503	7.456
35	447	7.796
40	381	8.257
45	323	8.733
50	279	9.156
60	221	9.828
70	185	10.342
80	173	10.535
90	150	10.947
100	140	11.146
110	130	11.360
120	117	11.663
130	109	11.868
140	107	11.922
150	104	12.000
160	96	12.235
170	89	12.453
180	84	12.619
200	74	13.000
240	66	13.316
250	61	13.543
270	53	13.948
300	46	14.357
325	43	14.552
400	37	15.000

As an example, the ball-mill product from the test on Homestake ore at minus 48 mesh contained 45.3 per cent cumulative on 150 mesh, and 54.9 per cent cumulative on 200 mesh.

$$n = 12 + \frac{50 - 45.3}{54.9 - 45.3} = 12 + \frac{4.7}{9.6} = 12.489$$

$0.1505 \times 12.489 = 1.880$
$3.906 - 1.880 = 2.026 = \log 106$
Median diameter of ball-mill product = 106 microns.

Table 3 has been computed from the following formula for screens not in the Tyler standard screen scale but which are commercially used:

$$n = \frac{\log 6664 - \log \text{opening in microns}}{\log 1.4141}$$

The figure 6664 microns is the theoretical opening of a 3-mesh screen, whose ordinal number is zero on the scale adopted.

When a screen analysis is given in the screens listed in Table 3 rather than in the Tyler standard screen scale, the value of n is computed from the formula:

$$n = n_1 + (n_2 - n_1)\frac{50 - \text{per cent cum. on } n_1}{\text{per cent cum. on } n_2 - \text{per cent cum. on } n_1}.$$

The median diameter is computed from n as before.

When more than 50 per cent cumulative of the sample passes through 200 mesh (or through the finest screen used) an extrapolation can be made, the probable accuracy of which decreases rapidly as the percentage of minus 200 mesh increases. It has been applied up to 75 per cent minus 200 mesh.

Considering first the case where the percentage through 200 mesh is less than 100 minus the percentage cumulative retained on 150 mesh,

$$n = 13 + \frac{\text{per cent through 200 mesh} - 50}{\text{per cent cum. on 200 mesh} - \text{per cent cum. on 150 mesh}}$$

When the percentage through 200 mesh is higher than in the example above, but less than 100 minus the percentage cumulative retained on 100 mesh,

$$n = 13 + \frac{\text{per cent through 200 mesh} - 50}{\frac{1}{2}(\text{per cent cum. on 200 mesh} - \text{per cent cum. on 100 mesh})}$$

A number of ores were tested for comparative grinding resistance by Lennox.[3] These were dry open-circuit tests, all ground for the same length of time, with the comparative values computed from the screen analyses of the mill products. They are open to the objection that the feed samples were all composite samples artificially adjusted to have the same screen analysis, thus destroying to some extent the representative character of the samples tested. The grinding resistance of the Portland ore was taken as unity; that of the Miami ore was found to be 0.70; and that of the Homestake was 0.63. These values agree very well with our grindability determinations at minus 100 mesh, the Miami ore being 1.1 per cent lower and the Homestake ore 1.5 per cent higher than our grindability results, but they are widely different from our results at any other mesh.

This appears to indicate that the grinding resistance of the ores in the Lennox scale, which are easier to grind than the Portland, should be less than the listed values when grinding coarser than minus 100 mesh, and greater when grinding finer than minus 100 mesh. It also serves to emphasize the necessity for making grinding tests at the mesh to be used in practice, where a reasonably accurate comparison is to be made to determine the relative grindability of any given ore as compared to any other ore.

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[3] Reference of footnote 1.

We believe that the close agreement at 100 mesh between results secured by Lennox with open-circuit grinding and ours obtained by closed-circuit grinding in this particular case are due largely to the fact that the three ores compared are relatively homogeneous in themselves as regards grindability of their component minerals.

Comparison of Wet and Dry Grinding

Wet grindability tests were made on the Portland and Miami ores in order to determine the relative production by wet and dry grinding. After grinding dry for a sufficient number of periods to bring the circulating load to equilibrium, sufficient water was added to make the ball-mill charge either 50 or 67 per cent solids by weight, and the grinding was continued wet. At the end of the period the mill discharge was screened wet, the oversize dried and screened dry through the screens, of the same mesh and the dry oversize weighed as the circulating load. Fresh feed and water were added and the charge returned to the mill for the second wet grinding period. This was continued until equilibrium was reached.

Table 4.—*Summarized Comparison of Wet and Dry Grinding*

Mesh	28	48	48	100	200
Ore	Portland	Miami	Miami	Miami	Miami
Per Cent Solids	50	50	67	67	67
Grindability: Dry, per cent	1.966	2.610	2.610	1.816	1.139
Wet, per cent	3.05	3.82	4.55	3.645	2.17
Wet ÷ Dry, per cent	155	146	174	201	190.5
Revolutions to grind unit volume mill feed $\frac{\text{Dry}}{\text{Wet}}$, per cent	159.5	148.5	180.5	203	186.8
Mill feed, n	4.268	4.931	4.931	4.931	4.931
Median diameter	1826	1454	1454	1454	1454
Circulating load, dry, n	4.108	5.916	5.916	8.466	10.935
Median diameter	1935	1031	1031	428.5	181
Circulating load, wet, n	3.995		4.777	7.365	10.827
Median diameter	2012		1538	617	188.7
Median diameter, $\frac{\text{Wet}}{\text{Dry}}$, per cent	104.0		149	144	104
n dry − n wet	0.113		1.139	1.101	0.108
Undersize in feed, per cent	19.8	18.1	18.1	11.8	7.5

The results obtained are listed in Table 4. They show that the net production in wet grinding at different meshes and dilutions varies from 146 to 201 per cent of that in dry grinding; also, that in grinding Miami

TABLE 5.—*Results of Grindability Tests*

Ore	Mesh	Grindability, Grams per Rev.	Rev. to Make Unit Volume	Median Diameter, Microns		
				Feed	Circ. Load	Product
Pure crystallized quartz	28	4.55	258	1650	1333	336
	48	2.72	432	1650	670	187
	100	1.523	772	1650	293	83.3
	200	0.878	1338	1650	167	41.7
Portland gold ore, Colorado	28	1.966	581	1826	1935	254
	48	1.640	696	1826	1454	151.6
	100	1.256	910	1826	680	80.5
	200	1.035	1103	1826	215	37.6
Miami copper ore, Arizona	28	3.370	341	1454	1650	254
	48	2.610	440	1454	1031	141.5
	100	1.816	634	1454	428	84.3
	200	1.139	1010	1454	181	35.0
Homestake gold ore, South Dakota	28	3.95	338	1675	1895	182
	48	2.95	453	1675	1350	106
	100	1.964	680	1675	544	63.2
	200	1.26	1060	1675	206	28.7
Bingham Canyon ore, Utah. Heavy sulfide, U. S. mines	48	5.90	318	1501	926	153
	100	2.99	630	1501	341	99.5
	200	1.854	1015	1501	197.5	39.8
Cons. Copper porphyry ore, Nevada	48	5.32	216	961	682	143
	65	3.63	317	995	473	101.3
	100	2.59	444	995	303	67.7
Tennessee copper ore, Tennessee	48	4.80	304	1333	802	165
	65	3.29	443	1333	527	119
Sherritt-Gordon ore, Canada	48	2.97	438	1425	980	168
	100	1.64	793	1530	372	76
Oolitic iron ore, Keradid, Morocco	35	2.97	438	1683	1556	212
	65	1.94	671	1683	887	122.5
Fertilizer rock	48	2.41	402	1725	902	168
	100	1.36	713	1725	396	68.3
Magnetite-apatite, Brazil	65	2.255	732	1425	538	110
	100	1.47	1122	1425	334	74
Mines de Bor–A, copper ore, Serbia	150	2.31	636	2210	1134	
	200	1.94	757	2210	889	
Noranda	48	1.38	935	1905	1864	81.5
	100	1.07	1205	1905	1678	
	200	0.83	1552	1905	1420	
Wright-Hargreaves	28	1.338	891	2073	2210	202
	48	1.24	961	1850	2070	117
	100	0.98	1215	1850	1840	68
	200	0.771	1545	2073	215	

ore the production is considerably higher at 67 per cent solids than at 50 per cent solids. The average grain diameter of the circulating load is larger in wet than in dry grinding.

It is hoped that further studies may be made to show the effect of other variables on the rate of grinding by a similar method. It is proposed that a large minus 6-mesh sample of an ore should be prepared and a grindability test made, preferably at about 100 mesh. When equilibrium has been reached any one of the following factors can be changed and the sample brought to equilibrium under the new conditions: (1) mill speed, (2) weight of ball charge, (3) size of balls, (4) use of shapes other than

TABLE 6.—*Results of Grindability Tests*

Ore	Mesh	Grindability, Grams per Rev.	Rev. to Make Unit Volume	Median Diameter, Microns		
				Feed	Circ. Load	Product
Wright-Hargreaves	28	1.338	891	2,073	2,210	202
Portland gold ore, Colorado		1.966	581	1,826	1,935	254
Miami copper ore, Arizona		3.370	341	1,454	1,650	254
Homestake gold ore, S. Dakota		3.95	338	1,675	1,895	182
Dwight-Lloyd sinter		4.01	394	1,712	1,510	312
Hualgayoc silver ore, Peru		4.27	402	1,625	1,655	259
Pure crystallized quartz		4.55	258	1,650	1,333	336
Red Cross gold ore, California		20.9	52.7	629	1,188	238
Oolitic iron ore, Keradid, Morocco	35	2.97	438	1,683	1,556	212
Iron ore, Belmonte, Spain		3.03	490	1,475	1,802	214
Wright-Hargreaves	48	1.24	961	1,850	2,070	117
Noranda ore		1.38	935	1,905	1,864	81.5
Portland gold ore, Colorado		1.640	696	1,826	1,454	151.6
San Fernando—B, Mexico		2.28	504	1,772	1,290	157.2
San Fernando—A, Mexico		2.31	474	1,865	1,184	153.3
Garnet, Georgia		2.40	519	1,350	1,243	143
Fertilizer rock		2.41	402	1,725	902	168
Miami copper ore, Arizona		2.610	440	1,454	1,031	141.5
Pure crystallized quartz		2.72	432	1,650	670	187
Homestake gold ore, S. Dakota		2.95	453	1,675	1,350	106
Sherritt-Gordon ore, Canada		2.97	438	1,425	980	168
Cons. Copper (limestone), Nevada		3.00	359	1,790	1,255	136.8
Shale, Quebec		3.50	317	1,350	883	139
Tennessee copper ore, Tennessee		4.80	304	1,333	802	165
Cons. Copper (porphyry), Nevada		5.32	216	961	682	143
Bingham Canyon ore, Utah		5.90	318	1,501	926	153
Zinc ore, Illinois		6.74	181	1,122		
Graphite ore, Alabama		9.26	102	719	507	191.4
Cast iron	65	0.0614	40,000	1,425	595	136.8
Britannia Mining copper ore, B		1.577	762	1,475	1,027	120
Pyrite in coal		1.61	737	1,917	1,700	93.5
Oolitic iron ore, Keradid, Morocco		1.94	671	1,683	887	122.5
Chelan copper ore, Washington		2.07	555	1,840	1,078	106
Magnetite-apatite, Brazil		2.255	732	1,425	538	110
Homestake gold ore, S. Dakota		2.546	524	1,675	1,030	77.2
Tennessee copper ore, Tennessee		3.29	443	1,333	527	119
Cons. Copper (porphyry), Nevada		3.63	317	995	473	101.3
Wright-Hargreaves	100	0.98	1,215	1,850	1,840	68.0
Noranda ore		1.07	1,205	1,905	1,678	
Portland gold ore, Colorado		1.256	910	1,826	680	80.5
Fertilizer rock		1.36	713	1,725	396	68.3
Magnetite-apatite, Brazil		1.47	1,122	1,425	334	74
Pure crystallized quartz		1.523	772	1,650	293	83.3
Dahlonega gold ore, Georgia		1.58	739	1,137	249	84.7
Sherritt-Gordon ore, Canada		1.64	793	1,530	372	76
Miami copper ore, Arizona		1.816	634	1,454	428	84.3
Homestake gold ore, S. Dakota		1.964	680	1,675	544	63.2
Ipo gold ore, Philippine Islands		2.01	587	1,180	284	84.3
Falconbridge nickel ore, Canada		2.405	668	1,440	356	73.5
Marquette iron ore, Michigan		2.50	608	1,770	1,060	33
Cons. Copper (porphyry), Nevada		2.59	444	995	303	67.7
Bingham Canyon ore, Utah		2.99	630	1,501	341	99.5
Victor Chem. slag, Chicago		4.15	682	553	237	92.6
Marquette iron ore (roasted), Michigan		6.80	222	637	318	43

TABLE 6.—(*Continued*)

Ore	Mesh	Grindability, Grams per Rev.	Rev. to Make Unit Volume	Median Diameter, Microns		
				Feed	Circ. Load	Product
Bong gold ore, Indo-China	150	1.31	1,070	1,196	222	
Mines de Bor—B, copper ore, Serbia		1.41	852	2,370	477	
Mines de Bor—A, copper ore, Serbia		2.31	636	2,210	1,134	
Mineral wool	200	0.678	1,040			
Wright-Hargreaves		0.771	1,545	2,073	215	
Noranda ore		0.83	1,552	1,905	1,420	
Pure crystallized quartz		0.878	1,338	1,650	167	41.7
Du Pont chem. iron slag		0.89	2,150	217	137	
Ilmenite sand		0.92	2,120	222	164	
Portland gold ore, Colorado		1.035	1,103	1,826	215	37.6
Ilmenite, Vanadium Corporation		1.08	1,582	284	146	
Miami copper ore, Arizona		1.139	1,010	1,454	181	35.0
Baguio ore		1.160	974	810	168	
Homestake gold ore, S. Dakota		1.26	1,060	1,675	206	28.7
Apatite, Vanadium Corporation		1.76	671	172.5	136.7	
Bingham Canyon ore, Utah		1.854	1,015	1,501	197.5	39.8
Mines de Bor—A, copper ore, Serbia		1.94	757	2,210	889	
Lead slag mass (brittle)		5.16	615	1,350	217	

TABLE 7.—*Grindability Scales by Weight and by Volume*

At 28 Mesh

	Weight[a]		Volume	
Portland gold ore, Colorado	1	1.00	1	1.00
Miami copper ore, Arizona	2	1.71	4	1.70
Homestake gold ore, S. C.	3	2.01	5	1.72
Dwight—Lloyd sinter	4	2.04	3	1.47
Hualgayoc silver ore, Peru	5	2.17	2	1.44
Pure crystallized quartz	6	2.32	6	2.25
Red Cross gold ore, California	7	10.60	7	11.00

At 48 Mesh

Portland gold ore, Colorado	1	1.00	1	1.00
San Fernando—B, Mexico	2	1.39	3	1.38
San Fernando—A, Mexico	3	1.41	4	1.46
Garnet, Georgia	4	1.46	2	1.34
Fertilizer rock	5	1.47	9	1.73
Miami copper ore, Arizona	6	1.59	6	1.58
Pure crystallized quartz	7	1.66	8	1.61
Homestake gold ore, S. Dakota	8	1.80	5	1.53
Sherritt-Gordon ore, Canada	9	1.81	7	1.59
Cons. Copper (limestone) Nevada	10	1.83	10	1.94
Shale, Quebec	11	2.14	12	2.19
Tennessee copper ore, Tennessee	12	2.93	13	2.28
Cons. Copper (porphyry) Nevada	13	3.24	14	3.22
Bingham Canyon ore, Utah	14	3.60	11	2.18
Zinc ore, Illinois	15	4.10	15	3.84
Graphite ore, Alabama	16	5.65	16	6.81

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[a] Numbers in sequence 1, 2, 3, etc., relate to order of grindability in decreasing resistance from Portland ore taken as unity.

TABLE 7.—(*Continued*)

At 100 Mesh

	Weight[a]		Volume	
Portland gold ore, Colorado	1	1.00	2	1.00
Fertilizer rock	2	1.08	6	1.28
Magnetite—apatite, Brazil	3	1.17	1	0.81
Pure crystallized quartz	4	1.21	4	1.18
Dahlonega gold ore, Georgia	5	1.26	5	1.23
Sherritt-Gordon ore, Canada	6	1.31	3	1.15
Miami copper ore, Arizona	7	1.45	10	1.43
Homestake gold ore, S. Dakota	8	1.56	8	1.34
Ipo gold ore, Philippine Islands	9	1.60	13	1.55
Falconbridge nickel ore, Canada	10	1.92	9	1.36
Marquette iron ore, Michigan	11	1.99	12	1.50
Cons. Copper, (porphyry) Nevada	12	2.06	14	2.05
Bingham Canyon ore, Utah	13	2.38	11	1.44
Victor Chem., slag, Chicago	14	3.30	7	1.33
Marquette iron ore (roasted) Michigan	15	5.46	15	4.10

At 200 Mesh

	Weight[a]		Volume	
Pure crystallized quartz	1	0.85	3	0.82
DuPont chem., iron ore	2	0.86	1	0.51
Portland gold ore	3	1.00	7	1.00
Ilmenite, Vanadium Corporation	4	1.04	2	0.70
Miami copper ore, Arizona	5	1.10	6	1.09
Homestake gold ore, S. Dakota	6	1.22	4	1.04
Apatite, Vanadium Corporation	7	1.70	9	1.65
Bingham Canyon ore, Utah	8	1.79	5	1.08
Mines—DeBor—A. copper ore, Serbia	9	1.87	8	1.46
Lead slag mass (brittle)	10	4.98	10	1.79

[a] Numbers in sequence 1, 2, 3, etc., relate to order of grindability in decreasing resistance from Portland ore taken as unity.

balls, (5) use of an uneven mill lining, (6) moisture in dry grinding, (7) dilution in wet grinding, (8) amount of circulating load. The list can be greatly extended.

It is to be noted from the above that the median diameter of the circulating load is larger when grinding wet than when grinding dry; that a variation in moisture content also affects the grindability; and that the variation is greatest for the grinding at the finer mesh.

Table 5 shows the results of a number of tests in which the grindability of an ore was determined at more than one mesh. The second column gives the mesh at which the test was made. The third column gives the "grindability" in grams per revolution, or the relative ease of grinding equal tonnages of the different ores to pass the specified mesh.

In the fourth column the numbers of revolutions required to produce 700 c.c. (unit volume) of net finished product are listed. These values were obtained by dividing the weight in grams of 700 c.c. of the minus 6-mesh broken ore by the net production in grams per revolution. They are inversely proportional to the relative ease of grinding equal volumes of the different ores to pass the specified mesh; in other words, they show the "grindability" on a volume basis.

The median diameters of the feed, the circulating load and the ball-mill product are given in the fifth, sixth and seventh columns.

The difference between the grindabilities of different ores on a volume basis is usually less than the grindabilities on a weight basis. This emphasizes the natural advantage accruing to a heavy ore in increased tonnage ground.

Table 6 shows the results obtained from a number of ores that were tested at only one mesh. They are arranged in the order of increasing grindability for each mesh. The ores listed in Table 5 are included for comparison.

Table 7 shows the grindability ratios for the various materials tested, arranged to show a comparison with Portland gold ore taken as unity, both on a weight and volume basis.

SUMMARY

A method has been developed to determine the relative ease of grinding, or grindability in closed circuit, for ores and other materials.

The method consists of a closed-circuit dry grinding test which is continued until equilibrium has been established.

In these tests a constant volume of ore, and a constant weight of balls are used. The total surface of the grinding media is also maintained approximately constant.

The results obtained show that the grindability ratios for different ores are greatest at the coarser meshes, and that they decrease as the finer meshes are approached.

The tests show that various materials exhibit marked differences in grindability at different meshes.

Comparisons of grindability should always be made at a specified mesh.

The relative grindabilities of several materials are tabulated, both on a weight basis and on a volume basis at several different meshes.

A method is described for evaluating a screen analysis by calculating the diameter of the median grain.

Crushing and Grinding Characteristics as Determined from Screen Analyses

By Fred C. Bond* and Walter L. Maxson,† Members A.I.M.E.

There is more information contained in a screen analysis than is ordinarily recognized, and there is an increasing need for methods of making this additional information easily available. As more comprehensive studies of crushing and grinding are made, and as the possibilities of decreased expense and increased extraction through a closer control of grinding are explored, methods of interpreting screen analyses assume an importance which they have not had heretofore.

The information commonly desired, which can be determined from a complete and carefully made screen analysis, includes: (1) the size distribution of the material passing the finest screen, (2) the presence of a hard grinding fraction, (3) the location of natural grain sizes, (4) the presence of different materials with markedly different grinding characteristics, (5) the presence and amount of finely divided material such as clay, which is merely unlocked in grinding, and (6) the total surface area of the ground product.

It is necessary to know the fundamental law of size distribution of a homogeneous material in order to correctly resolve a screen-analysis curve, and to understand the variations and anomalies that a sample may exhibit when compared with the ideal curve.

This fundamental law was developed by Gaudin,[1] who showed that when the logarithm of the per cent weight retained on any screen and passing the next larger screen in the Tyler standard screen scale was plotted against the logarithm of the screen aperture the resulting curve for the finer sizes of a homogeneous crusher or grinding-mill product would be a straight line.

Gross and Zimmerley[2] experimentally confirmed Rittinger's law, which states that the useful work done in crushing a homogeneous material is directly proportional to the new surface area produced; and showed that in the larger screen sizes resulting from impact crushing a

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[1] A. M. Gaudin: An Investigation of Crushing Phenomena. *Trans.* A.I.M.E. (1926) **73**, 253.

[2] J. Gross and S. R. Zimmerley: Crushing and Grinding Studies of Quartz. U.S. Bur. Mines *Rept. of Investigations* 2880 (1928).

large proportion of the total surface exists as cracks or fissures. They called this "interior surface," which, of course, cannot be measured directly by screen analysis. They also determined that 55,785 ergs of useful work are required to produce one square centimeter of new quartz surface.

Weinig[3] used screen ordinal numbers instead of the logarithms of the screen apertures, and amply confirmed Gaudin's distribution law by screen analyses and by sedimentation balance tests and microscopic grain counts of the minus 200-mesh material. He also determined that the lower limit of an ordinary ore grind is approximately one micron, and that little or no colloidal material is produced that had not existed in the colloidal state in the ore prior to grinding.

The need for a simple and practicable method of applying this distribution law to screen analyses of products from different crushing and grinding machines, to secure information about the work of the machines and the character of the material ground, led to the development of the procedure described in this paper.

We have chosen to use the screen ordinal numbers as selected by Weinig, according to which a 200-mesh screen has an ordinal number of 41; a 150-mesh screen, an ordinal of 42; a 100-mesh screen, an ordinal of 43, etc. There is need for uniformity in the ordinal numbers assigned to screen sizes, and this system is practical in that positive numbers can be assigned to any conceivable particle size concerned in crushing and grinding.

If d is the average grain diameter in microns (or thousandths of a millimeter) of particles retained on any screen n and passing the next larger screen $n + 1$, then

$$d = a\sqrt{2}^{n-1} \qquad [1]$$

where a is a constant such that[4]

$$\log a = \bar{5}.9285948$$

For screens in the Tyler standard screen scale n is always a whole number.

The method of plotting screen analyses is shown in Figs. 1, 2 and 3. Ordinary cross-section paper is used, with the ordinal numbers n as abscissas and the log per cent weight as ordinates. Ordinal number $n = 41$ is chosen as the Y axis, and $X = \frac{n - 41}{10}$.

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[3] A. J. Weinig: A Functional Size Analysis of Ore Grinds. Colo. School of Mines *Quarterly* (1933) **28**, No. 3.

[4] All logarithms employed in this article are to the base 10.

If the screen analysis of a homogeneous ground product is plotted it will follow the curve as shown in Fig. 1. The straight line

$$y = mx + b \tag{2}$$

is called the distribution line. Its Y intercept b is the logarithm of the per cent weight passing 150 mesh and retained on 200 mesh, and its slope m is the most important single quantity derived from the graph, since it is characteristic both of the material being broken and the type of machine and operating conditions. According to the distribution law the curve continues as a straight line with decreasing values of n into the colloidal size range. In general agreement with Weinig's results the inception of the colloidal size range is taken here as the limit of the average ore grind. At this point n equals 28, and the average grain diameter is 0.9829 microns.

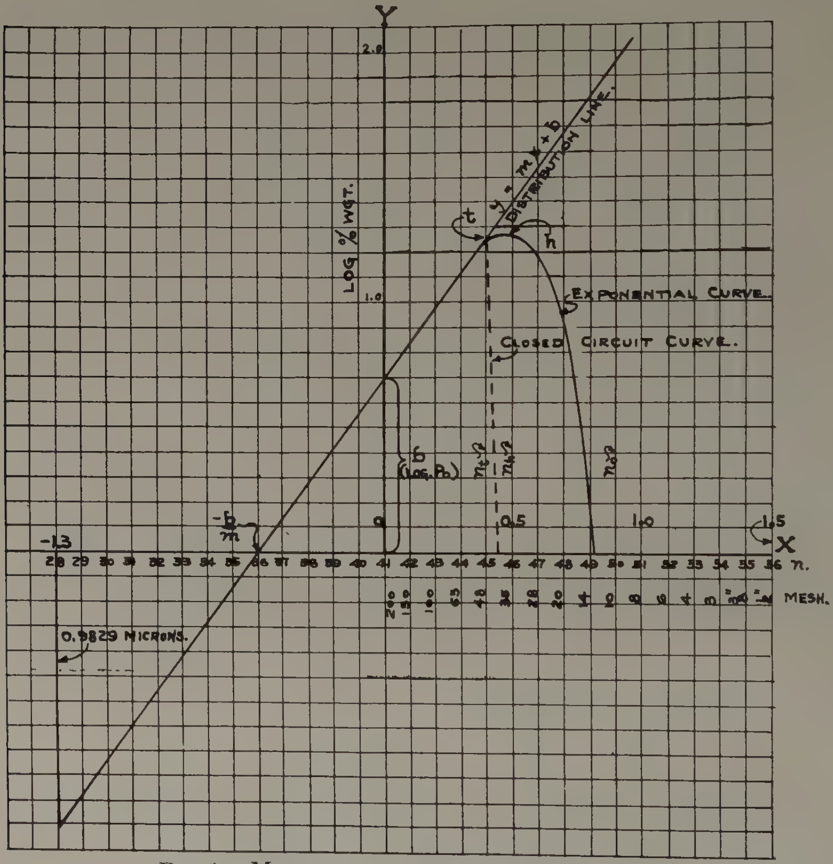

FIG. 1.—METHOD OF PLOTTING SCREEN ANALYSES.

It sometimes happens with heterogeneous materials, and particularly with mixtures of two or more materials, that more than one distinct distribution line is present. In such cases the line that extends to the colloidal range is called the primary distribution line and the one that first crosses it as the coarser sizes are approached is called the secondary line. Additional lines can be present, depending upon the character of the material.

The ordinal number of the finest screen passing 100 per cent of the sample is designated as n_0; the number of the screen that retains the largest per cent weight is n_h; and that of the screen where the plotted curve becomes tangent to the primary distribution line is n_t. The curve follows the primary distribution line from n_t to n_{28}.

Screen analyses are made by the wet-dry method; the weighed sample is washed with a stream of water on a 200-mesh sieve, usually used with a coarse protecting sieve, and the oversize is dried, weighed, and screen-analyzed dry. By this method the retention of adhering fines to the larger grains and the blinding of the finer screens are minimized. Only a few trials are necessary to convince one of the superior accuracy of this method, and we have found that it is often difficult to properly locate the distribution line on screen analyses made without the wet split. The various screen samples are also more suitable for immediate microscopic examination and for analysis. The material passing the 200-mesh sieve wet is designated as minus 200-mesh slimes, and that retained wet but passing it dry is called minus 200-mesh sands. However, these two products are ordinarily united in calculations of screen analyses.

Screen analyses made with screens other than those in the Tyler standard screen scale can be resolved. In such cases it is necessary to compute the value of n to three decimal places for each screen used. The value of d in microns for each screen is computed as the average of the screen apertures in microns of the retaining screen and of the next larger screen used. The corresponding value of n is found from:

$$n = 1 + \frac{\log d - \overline{5}.9285948}{0.150515} \qquad [3]$$

It is evident that the minus 200-mesh material delineated by the distribution line down to n_{28} represents all of the finer particles produced as broken fragments of larger particles in the process of crushing or grinding. If we let G represent the per cent weight of this material produced directly by grinding, F represent the per cent weight of the total minus 200-mesh material as determined from screen analysis, and U the per cent weight of any unlocked material such as clay which existed in the divided state in the ore before grinding, and which we will call the *unground fines*, then:

$$U = F - G \tag{4}$$

When the distribution line in Fig. 1 is extended to n_{28} it can be shown that

$$G = \frac{4.343P_0}{m}\left[1 - \frac{1}{\text{antilog }(1.3m)}\right] \tag{5}$$

where P_0 is the per cent weight passing 150 mesh and retained on 200 mesh, or the antilogarithm of b.

When the value of G is computed for any particular grind and substituted in equation 4, U can be determined. Thus the percentage of unground fines existing in an ore and unlocked at any particular grind can be calculated. This material is considered to be chiefly colloidal or semicolloidal.

It sometimes happens in coarse breaking that the grind limit does not extend down to 0.9829 microns, or to n_{28}, in which case G as calculated from equation 4 is larger than F. It then becomes necessary to compute the actual grind limit, which we will call n_L. It can be shown that:

$$n_L = 41 + \frac{10}{m}\log\left(1 - \frac{Fm}{4.343P_0}\right) \tag{6}$$

and the actual grind limit in microns is found by interpolating n_L in column 3, Table 1, or by substitution of n_L for n in equation 1.

After the equation of the distribution line has been determined the total surface area of the minus 200-mesh material produced in grinding can be computed. The total surface area of a solid cube 1 cm. on a side, when broken into cubes which measure d microns on a side, is 6 divided by d sq. m. From equation 1 the total surface area S_n of 1 c.c. of solid material which all passes screen $n + 1$ and is retained on screen n is expressed by

$$S_n = \frac{6}{a\sqrt{2^{n-1}}}\text{ sq. m.} \tag{7}$$

It can be shown that the total surface area of 1 c.c. of solid material broken to form spheres of diameter d will be equal to that of an equal volume broken to form cubes d microns on a side. It appears probable that the total surface area of the irregular grains produced by grinding which pass screen $n + 1$ and are retained on screen n will be somewhat greater than that of either cubes or spheres, but the ratio should be fairly constant for the different screen sizes in all cases where the interior surface is not disproportionate. This was confirmed by Gross and Zimmerley, who determined experimentally that the ratio of actual to theoretical surface area of Ottawa sand over a range of 20 to 200 mesh was almost constant at an average value of 1.34 to 1. We have made measurements on a jaw-crusher product which show that there is a slight increase in the ratio of actual to theoretical external surface, amounting to about 5 per cent, in the range from 20 to 3 mesh. At coarser meshes the ratio increased more rapidly, probably because of the tendency of the jaw crusher to form slabs.

In any case the error as applied to ground products is small enough to be safely disregarded for the present. The percentage error introduced by the presence of interior surface in coarse crusher products is greater, and comparisons of the work done in crushing with that done in grinding should be made with diffidence until a correction factor can be found.

If a sample of 100 c.c. of solid material is taken, the surface area S in square meters for any value of n is given by the equation:

$$S = 0.06745P_0 \times \text{antilog}\left[\left(\frac{n}{10} - 4.1\right)(m - 1.50515)\right] \qquad [8]$$

and the total surface area S_G of the noncolloidal minus 200-mesh material is:

$$S_G = \frac{0.292907P_0}{1.50515 - m}\left\{\text{antilog}\,[1.3(1.50515 - m)] - 1\right\} \qquad [9]$$

In cases where the grind limit n_L has been found to be above $n = 28$, the computed value of $\frac{41 - n_L}{10}$ should be substituted for 1.3 in equation 9.

It can be seen, from an inspection of equation 8, that where $m = 1.50515 = 10 \log\sqrt{2}$ the total surface area contained between any two adjacent values of n will be independent of the actual values of n; for example, the total surface contained between n_{41} and n_{42} will be the same as that contained between n_{28} and n_{29}, or between any other two adjacent values of n.

When m is greater than 1.50515, the total surface area of the material retained between any two adjacent values of n decreases as n decreases, and when m is less than 1.50515 it increases as n decreases. This relationship is valuable as an indication as to what proportion of the total work done in crushing or grinding has been expended in producing minus 200-mesh material.

In Table 1 ordinal numbers are listed in column 1, and the corresponding meshes in column 2. Column 3 lists the average diameter in microns of grains passing screen $n + 1$ and retained on screen n, as calculated from equation 1; column 4 contains the corresponding surface areas from 1 c.c. of solid material, as calculated from equation 7. In column 5 the values from column 4 are suitably rounded off for quick use with a slide rule.

All calculations are ordinarily made on a slide rule. After the weights of the various screen fractions are obtained, the per cent weight and the log per cent weight are both read and recorded, then the per cent weight is multiplied by the value given in column 5 of Table 1 for that screen size, and the product recorded. This represents the equivalent surface retained on screen n and passing screen $n + 1$ from 100 c.c. of total sample.

TABLE 1.—*Values Calculated from Equations*

1 n	2 Mesh	3 d (Microns)	4 S in M^2 per C.C. Solids	5 S (Slide Rule)
	Inch			
60	2	64,360	0.00009315	0.0000931
59	1½	45,520	0.00013172	0.0001317
58	1	32,180	0.00018629	0.0001863
57	¾	22,760	0.00026345	0.0002635
56	½	16,090	0.00037258	0.000373
55	⅜	11,376	0.00052691	0.000527
	Mesh			
54	3	8,052	0.00074516	0.000745
53	4	5,694	0.00105382	0.001054
52	6	4,026	0.00149032	0.001490
51	8	2,847	0.00210764	0.002108
50	10	2,013	0.00298064	0.002980
49	14	1,423	0.00421528	0.00422
48	20	1,006.5	0.00596128	0.00596
47	28	711.7	0.00843056	0.00843
46	35	503.2	0.01192256	0.01192
45	48	355.8	0.01686112	0.01686
44	65	251.6	0.02384512	0.02385
43	100	177.9200	0.03372224	0.0337
42	150	125.8112	0.04769024	0.0477
41	200	88.96000	0.06744448	0.0675
40	270	62.9056	0.09538048	0.0954
39	400	44.4800	0.13488896	0.1349
38	560	31.4528	0.190761	
37	800	22.2400	0.269778	
36	1,125	15.7264	0.381522	
35	1,600	11.1200	0.539556	
34	2,250	7.8632	0.763044	
33	3,200	5.5600	1.079112	
32	4,500	3.9316	1.526088	
31	6,400	2.7800	2.158224	
30	9,000	1.9658	3.052176	
29	12,800	1.3900	4.316448	
28	18,000	0.9829	6.104352	

Typical Screen-analysis Calculation

The screen analysis given in Table 2 is that of a gold ore from Montana which had been passed through a gyratory crusher set at ⅜-in. opening. The results are plotted in Fig. 2.

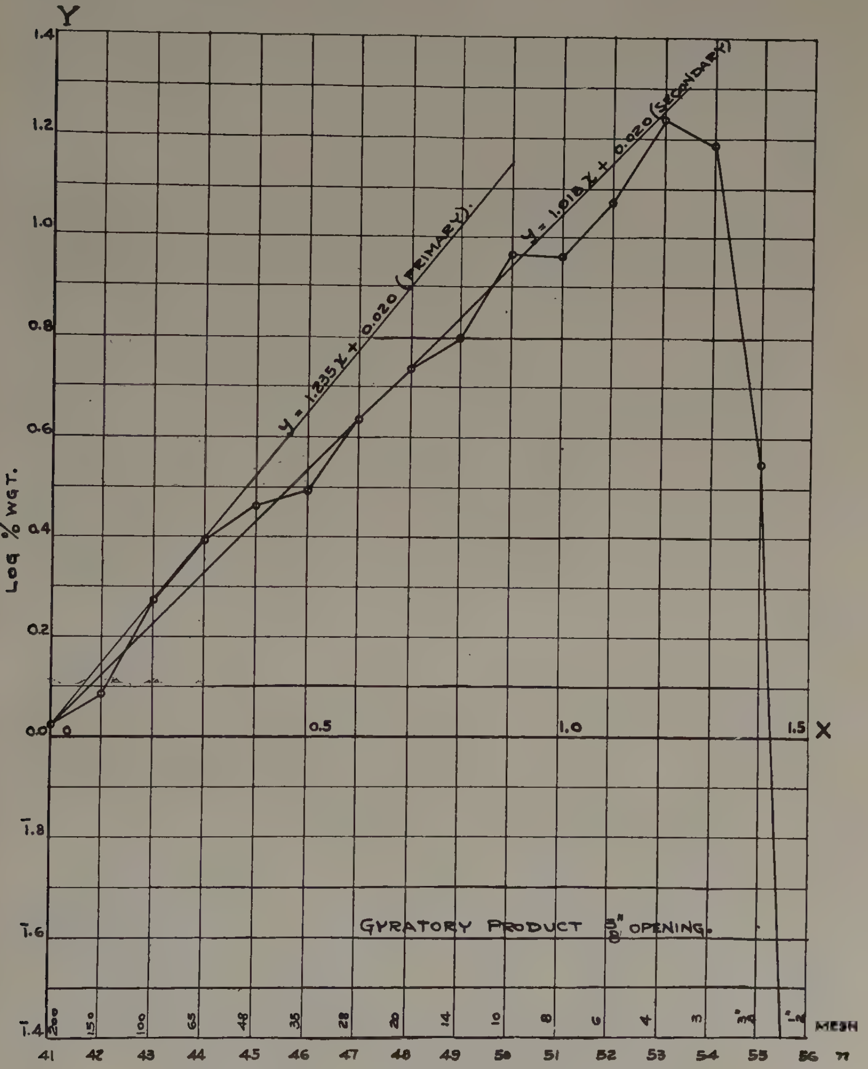

Fig. 2.—Screen analysis of Montana gold ore.

After the curve has been plotted, distribution lines are drawn to indicate the dominant trends, and the departures of the curve from these lines are highly significant. A departure above the line, or hump in the curve, shows the location of a natural grain size, at which point more than the usual amount of material is produced. It frequently happens that substantial unlocking of the minerals being ground occurs at this point. The natural grain size is a remarkably persistent characteristic. If the screen analyses are carefully made, it will be found at the same screen size for the same material when ground to widely different degrees of fineness, and even when different machines are used. However, it sometimes happens that the interior surface produced in crushing may obscure it. Samples have been analyzed which exhibited a distinct grain size in the exponential portion of the curve, lying to the right of *h*. If a depression persists at the same point with different materials it is well to examine that particular screen for breaks or wear.

TABLE 2.—*Screen Analysis of Montana Gold Ore*

Mesh		Per Cent Wt.	Cum. Per Cent	Log Per Cent Wt.	*n*	Surface, Sq. M
Inch						
On	½	0	0		56	
	⅜	3.53	3.53	0.548	55	0.00186
Mesh						
	3	15.46	18.99	1.190	54	0.01151
	4	17.40	36.39	1.242	53	0.01838
	6	11.98	48.37	1.079	52	0.01786
	8	9.12	57.49	0.960	51	0.01921
	10	9.17	66.66	0.962	50	0.02740
	14	6.31	72.97	0.800	49	0.0266
	20	5.40	78.37	0.733	48	0.0322
	28	4.32	82.69	0.636	47	0.0364
	35	3.11	85.40	0.493	46	0.0371
	48	2.89	88.69	0.461	45	0.0487
	65	2.43	91.12	0.386	44	0.0580
	100	1.85	92.97	0.267	43	0.0624
	150	1.21	94.18	0.084	42	0.0578
	200	1.05	95.23	0.020	41	0.0706
Through 200		4.77	4.77			
						0.52602
		100.00				

Primary grindability line $y = 1.235x + 0.020$

$m = 1.235$

$P_0 = 1.05$

$G = 3.60$ per cent (by substitution in equation 5).

$U = 4.77 - 3.60 = 1.17$ per cent unground fines.

$S_G = 1.414$ (by substitution in equation 9).

$S_T = 1.414 + 0.526 = 1.940$ sq. m. total surface area per 100 c.c. solid ore.

The material in an ore that has the greatest resistance to grinding will have the steepest distribution line, and the point where two distribution lines meet is important as giving an indication of the amount of each material present. In comparing different ores it does not follow that the hardest one to grind always has the steepest distribution line. Sediments and loosely consolidated materials are usually more heterogeneous, and exhibit more distribution lines, than igneous rocks.

The presence of a hard grinding fraction is shown by a hump extending above the distribution line at h, or at the screen retaining the largest percentage by weight. This may be caused by the presence of a small amount of a hard grinding material, but it is often the result of crushing or grinding conditions. It is present in a crusher product where a considerable amount of flat slabs is formed. In this case the hump is usually at a screen size larger than that of the crusher opening. When it exists in the screen analysis of a ground product, which is known not to contain any abnormally hard grinding material, it is a direct indication that the largest pieces in the mill feed are not being broken efficiently but are being worn by attrition, and usually heavier grinding media should be used or the mill speed increased.

The most important feature in the interpretation of the curve is the correct determination of the slope m of the primary distribution line. The example given in Fig. 2 was chosen to illustrate a condition that often arises in this regard. Although the secondary distribution line, with a slope of 1.018, appears as the dominant line, the primary line with a slope of 1.235 should be used. Serious errors result from selecting the point of tangency too far to the right, and in many cases it is preferable to take the slope from n_{43} to n_{41}, or from 100 to 200 mesh. In making comparisons as between different grinds on the same ore, it is always preferable to select the same value of n as the point of tangency of the primary distribution lines. Errors in the slope chosen are shown by wide variations in the computed values of the *unground fines* in different tests on the same ore. The percentage of unground fines should usually be small and should increase only slightly with finer grinding. If the variation is large with the same values of n_t it indicates that the screen analyses were made without a wet split, or are otherwise in error. Grinding naturally shows a considerable increase in the percentage of unground fines over crushing, since the attrition in grinding frees the fine material.

The unground fines are believed to exist chiefly as cementing material, and grinding to below the smallest natural grain size of the ore unlocks most of them. The amount present in ordinary siliceous ores is usually below 2 or 3 per cent, but when very soft material is ground, and particularly when a large amount of clay is present, it may rise to 10 per cent or more. It is not necessarily all colloidal material. It includes excess material unlocked at any natural grain size below 200 mesh, and some grinding may take place on these "excess particles." However, this error cannot be avoided without resorting to size distribution measurements below 200 mesh, and, according to measurements already made, the error is usually unimportant.

In ore grinding it is ordinarily desired to break down to the point of satisfactory unlocking. Breaking beyond this point represents work that is largely or completely wasted, and it follows therefore that the most efficient grinding is done when the slope of the primary distribution line m is at a maximum. Every possible effort should be made in ore grinding to regulate the conditions so as to increase the value of m. The average value found from tests on a number of different ores was around 1.5, although different ores crushed or ground in the same manner showed a variation in the value of m of from below 1.0 to above 2.0.

An increase in the amount of interior surface results in an increased value of m, since the interior surface is largely concentrated in the larger sizes; and a crushing machine is not credited with the interior surface produced when Rittinger's law is applied to screen analyses. For this reason it is difficult to correctly proportion the work done in crushing and in subsequent grinding, although it is safe to say that the portion of the total work done in crushing is always underestimated when computed from screen analyses. In grinding mills, and in all cases where attrition plays an important part, the ratio of interior surface to total surface is relatively small and is approximately constant for the different screen sizes, so that direct comparisons of the computed new surface areas are justified.

Comparative tests made on the same ores show that m is at a maximum in roll crushing, is somewhat less for gyratory and jaw crushers, and is considerably less for rod-mill and ball-mill products.

A number of comparative open and closed-circuit tests were made, and in every case the value of m was substantially higher in closed circuit, which proves that closed-circuit operation results in a decrease in the power loss due to overgrinding. Comparative wet and dry-grinding open-circuit tests were made, and gave approximately the same values for m, although the total new surface area produced in wet grinding was somewhat greater than that produced in dry grinding.

The results of tests made on a hard grinding ore which contained considerable clay are plotted in Fig. 3. The gyratory-product curve shows a characteristic hard grinding fraction hump at h, natural grain sizes at 10 mesh and 100 mesh, and two distribution lines, which cross at 20 mesh.

The roll-product curve shows a hump at 10 mesh, for which the natural grain size is partly responsible; a slight natural grain size at 100 mesh; and two distribution lines, which cross at 20 mesh. All of the distribution lines are steeper than in the gyratory product.

The ball-mill discharge from the standard grindability test shows an extremely large hard grinding fraction, or hump at h. It was evident that the balls used were too small to break the larger pieces efficiently.

The ball-mill discharge from the grindability test using 2-in. balls shows no hard grinding fraction, thus proving that the balls used were large enough for the efficient breaking of any particles present. The slope of the distribution line is evidently too great, owing to the over-

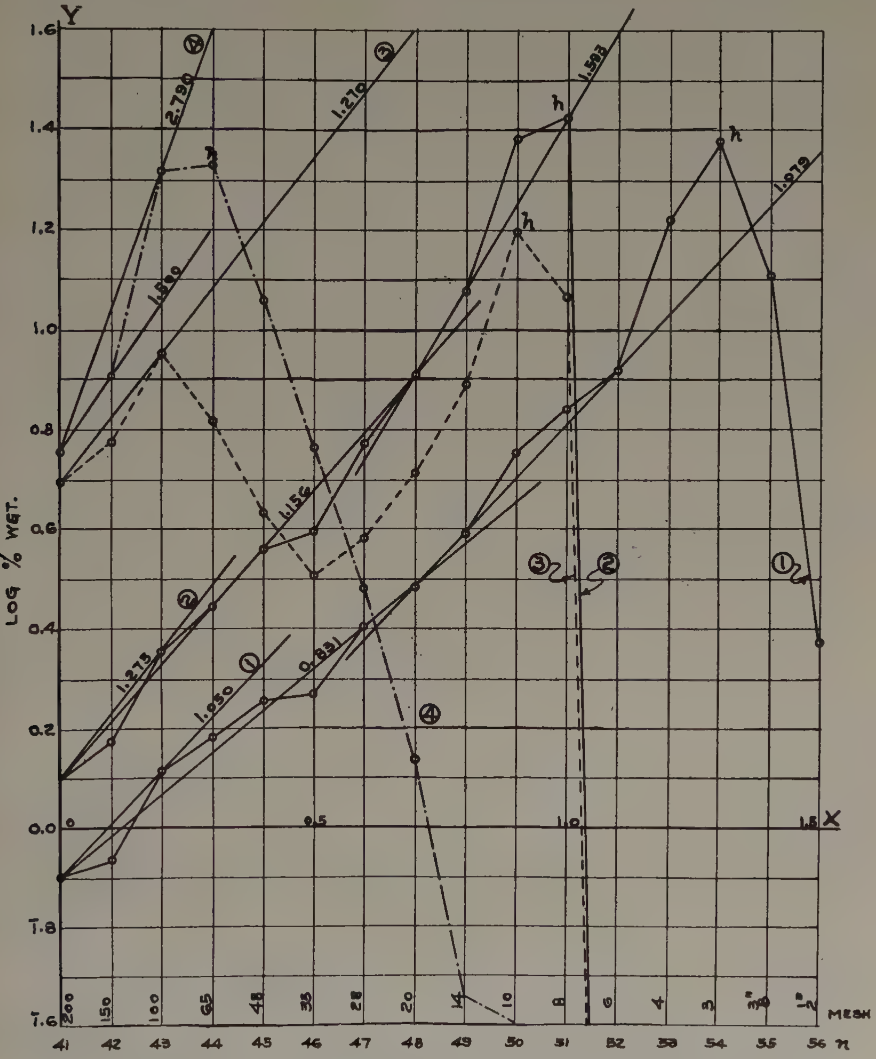

FIG. 3.—RESULTS OF TEST ON HARD GRINDING ORE CONTAINING CONSIDERABLE CLAY.

Curve 1 represents product of gyratory crusher set at 3/8-in. opening.

Curve 2, product of roll crushing in closed circuit with screen to all through 6 mesh.

Curve 3, ball-mill discharge from standard closed-circuit grindability test at 100 mesh, using balls ranging from 1½ to ¾ in. in diameter.

Curve 4, ball-mill discharge from grindability test similar to that of curve 3, except that balls were 2 in. in diameter.

Slopes of distribution lines are given; primary lines are tangent at n_{43}.

crowding at 100 mesh, and in this case it would be preferable to use the slope from 150 to 200 mesh.

The practical application of these data led to a reduction in the size of mill feed, and to the use of larger balls in the mill.

Standard closed-circuit tests were made on quartz at three different meshes by a method previously published,[5] and the new surface area produced was computed by equation 9. The ratio of actual to theoretical surface area was taken as 1.34 to 1, in accordance with Gross and Zimmerley's results. If their value for the surface energy of quartz (55,785 ergs) is increased in this proportion the surface energy becomes 75,000 ergs per square centimeter of new surface when computed from screen analyses. The useful work done per revolution of the mill in these tests was computed to be 3.88 joules at 28 mesh; 3.64 joules at 48 mesh; and 4.03 joules at 100 mesh. The average value was 3.85×10^7 ergs of useful work done per revolution.

The surface energy of any ore can be computed from a standard grindability test using this average value and if the power consumption in kilowatt-hours per ton and the screen analyses of the mill feed and product are available, it is possible to compute the absolute power efficiency obtained in grinding.

The work input to the mill in ergs per square centimeter of new surface produced is

$$\text{Ergs per sq. cm.} = \frac{397{,}000 \times \text{sp. gr.} \times \text{kw-hr. per ton}}{\text{New surface produced}} \qquad [10]$$

where the new surface produced is measured in square meters per 100 c.c. of solids ground. The surface energy as determined from a grindability test divided by the work input in ergs per square centimeter gives the net power efficiency obtained in grinding.

In computing the surface area of the mill product it is preferable to use the screen analysis of the mill discharge rather than that of the classifier overflow, since there will be less crowding in the finer screen sizes, and the value of m will be more accurate. Of course, the amount of the circulating load must be known.

Several such computations have already been made, but in most cases the reported screen analyses of the mill feed and product were apparently made without the wet split, and the values of m are in error. However, the calculated net power efficiency was generally higher than had been anticipated, and in one instance averaged around 10 per cent.

It should be emphasized that the surface energy of an ore, expressed as the number of ergs required to produce one square centimeter of new surface, is not directly proportional to the grindability, or to the relative ease of grinding, at any specified mesh. The characteristic slope m of the distribution line varies widely with different ores, and an ore with a flat distribution line may be very hard to break to any specified mesh and yet have a lower surface energy than an easier grinding ore. For instance, the hardest grinding ore ever tested in the laboratory had a grindability of 0.832 grams per revolution at 100 mesh, or 54.6 per cent of that of quartz, and yet its surface energy was 60,400 ergs per square centimeter, or 80.5 per cent of that of quartz. This is explained by the fact that the value of m was 1.270, as compared with 1.945 for quartz.

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[5] W. L. Maxson, F. Cadena and F. C. Bond: Grindability of Various Ores. See page 130, this volume.

The primary requirement in ore grinding is to break to a specified size, and *the practical efficiency* obtained is a measure of the work necessary to grind to this size. The amount of work lost in overgrinding depends upon the value of m, and for this reason comparisons between different ores, based upon the absolute power efficiency obtained in grinding, as computed from surface energies, do not yield relative practical efficiencies.

The only satisfactory method yet found for comparing practical efficiencies obtained in grinding two different ores having different values for m is to make laboratory grindability tests at the sizes to which the ores are ground in practice, and to then compare these relative grindabilities with actual operating power required in kilowatt-hours per ton.

While comparisons of this kind are of great interest to the industry, the average operator is primarily most concerned with means and methods whereby the grinding efficiency can be improved in his own plant. Such improvements may be expected only in those cases where the operator is prepared to make a systematic study of operating variables. The effect of changes in these variables can be correlated for easy comparison by developing the distribution-line curves for each set of conditions. Most important in interpreting such curves is a study of (1) the slope m of the distribution lines, (2) the presence of hard grinding fractions, (3) natural grain size and (4) new surface produced.

SUMMARY

Ordinal numbers are assigned to the screens in the Tyler standard screen scale, and the logarithms of the per cent weight retained are plotted against the ordinal numbers.

The resulting screen-analysis curve is a straight line for the finer screen sizes, and is called the "distribution line." The line extends to the colloidal size range, which is taken as the limit of the ordinary ore grind.

If the material is not homogeneous there will be some variations from this line.

The slope of the distribution line depends upon the character of the ore and upon the grinding conditions. An increase in the slope shows a decrease in the power lost in overgrinding.

The presence of a hard grinding fraction, the suitability of the grinding media to the size of the feed, and the existence and location of natural grain sizes, are shown by variations of the screen analysis curve from the distribution lines.

Equations are given by which the percentage of unground fines and the new surface area produced in grinding can be computed. Examples of the calculations and screen-analysis curves are included.

The practical efficiency in grinding depends upon the power used in breaking to a certain specified size, and comparisons of computed absolute power efficiencies obtained in grinding different ores are usually not concordant with the practical efficiencies.

Fine Grinding and Classification

By J. V. N. Dorr* and Anthony Anable,† Members A.I.M.E.

When separate treatment of sand and slime by cyanidation was the rule rather than the exception, as now, and when gravity concentration, rather than flotation, was the accepted method of dressing copper, lead, zinc and other base ores, classification was a separate step in ore treatment. Today, however, the picture is much different. The trend toward all-slime treatment in cyanidation and flotation has made fine closed-circuit grinding essential, and the mechanical classifier is of equal importance with the mill in the preparation of an ore for its treatment. Actually, a mill and classifier are a unit, a variation in either affecting the desired results.

In developing this thesis—the mutual interdependence of the rotary mill and the classifier in modern fine-grinding practice—it would be well to consider first the different types of classifiers and mills in use today, their scope and limitations and the factors governing their selection. From such a basis as this a logical approach may be made to modern closed-circuit fine grinding, the evolution of multiple-stage grinding practice, the relation of the classifier to the mill and, finally, the trend to higher and higher circulating sand loads in the mill-classifier circuit. Such data as are germane to the subject will be given to support the thesis advanced.

MECHANICAL CLASSIFIERS

Theory.—Stokes' law, applicable to the uniform slow motion of a rigid sphere in a viscous fluid of infinite extent, states that the velocity of a particle settling under the influence of gravity is a function of the square of its radius:

$$V = \frac{2}{9}\,\frac{g(d_s - d_e)\gamma^2}{S}$$

Where V = velocity of particle in cm. per sec.
g, acceleration due to gravity
d_s, density of particle
d_e, density of fluid
γ = radius of particle
S = viscosity of fluid

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† Engineer, The Dorr Co. Inc., New York, N. Y.

The limitations of the Stokes formula render it of little practical importance, because the particles must be perfect spheres, which they never are; the fluid must be of infinite extent, which is impossible; and the fluid must be homogeneous. Furthermore, it has been shown that even under theoretical conditions, Stokes' law applies only in the range of 200 mesh and finer, because on sizes ranging between 20 and 200 mesh the velocity varies with a power less than the square of the radius, being as the ½ power of the radius at the coarser end.

Stokes' law, however, serves a useful purpose in showing that: (1) the relative falling velocities of particles of the same specific gravity are functions of their sizes, the larger the particle the greater the settling rate; (2) the falling velocity decreases both with the density and viscosity of the fluid; and (3) the shape of the particle affects its falling velocity, spherical particles having the greatest velocity and thin, flat ones probably the least.

Practice.—In practice a vessel is provided in which the particles may settle roughly according to Stokes' law. In this vessel there is placed a mechanically operated means for setting up a counter-force, opposed to settlement, and for removing the settled solids. Provision is made for controlling the point of separation by varying the slope of the bottom of the vessel, by changing the speed of the mechanical element and by varying the viscosity and density of the pulp.

Mechanical classifiers may be grouped roughly into four types. The first two—the reciprocating rake Dorr classifier and the Dorr bowl classifier—are by far the most frequently used today. The Dorr classifier is a single-stage machine making separations in the 10 to 100-mesh range; the Dorr bowl classifier is a two-stage machine making separations in the finer range of 65 to 325 mesh. The Akins classifier, developed shortly after the Dorr, is of the revolving spiral type. The drag flight, or Esperanza type, employs a belt to which drags or flights are attached for securing the desired motion. The latter is seldom used today.

The point at which a separation is made in a mechanical classifier is determined by three factors; rake speed, overflow dilution and the slope of the tank bottom. Broadly speaking, the greater the rake speed, the lower the dilution and the steeper the slope, the coarser is the separation, and vice versa. With identical speed, dilution, slope, separation and ore, both overflow and raking capacities are proportional to the width of the classifier tank. Furthermore, the finer the separation is, the finer is the coarsest particle in the rake product, and consequently the slower must be the speed of the rakes and the smaller must be the slope of the tank to advance this fine sand up the sloping bottom. Both of these have the effect of reducing capacity.

HYDRAULIC CLASSIFIERS

Hydraulic classifiers range from simple V-shaped launders with a multiplicity of shallow settling pockets for the discharge of as many roughly sized products as desired, to the more elaborate and efficient units of the Fahrenwald type, equipped with pockets having constriction plates and jets of water for controlling the character of products collected in each pocket. The material in each pocket is hydraulically maintained in a condition known as "teeter," corresponding to true hindered settling, the particles arranging themselves not according to size but according to their hindered-settling ratios, which are functions of both specific gravity and particle size. Thus in practice, one pocket may contain particles small in size but of great density alongside of others large in size but of light weight. Obviously, both have the same hindered-settling rates. A discharge of particles having the same hindered-settling ratio is ideal, of course, as feed to a jig or a table, because the forces seeking to discard the light gangue operate effectively on its large particles and to the exclusion of the small, heavy particles of valuable mineral.

FACTORS GOVERNING A CHOICE OF CLASSIFIERS

A perfect classifier might be defined as one that would take a feed and separate it into two portions, the finer of which contained no particles coarser than a given size and the coarser of which contained no particle finer than the given size. On a homogeneous material this is theoretically possible with hydraulic classifiers, in which the coarse particles are forced to descend through a rising column of fresh or "hydraulic" water. On a heterogeneous material it is impossible to make the separation in one step. Nonhydraulic classifiers, in which all the water enters with the feed, are theoretically unable to make a complete clean separation, even on homogeneous materials. The fine portion can be made quite free from oversize, but the coarse portion always contains more or less fine material.

In spite of this fact, the mechanical, nonhydraulic classifiers are used most extensively because of their capacity, flexibility, continuous operation, and uniformity of results. In addition, they require little attention and give a well drained coarse portion or sand. Furthermore, the mechanical types of nonhydraulic classifier give a coarse portion that is freer from fines than one would expect from theory. This is due to the agitation and draining that take place in the course of disposal of the coarse portion. Advantages and disadvantages of mechanical and hydraulic classifiers are listed in the accompanying table.

Mechanical Classifiers

ADVANTAGES	DISADVANTAGES
Theory is well understood.	Because of the extremely slow settling rate of particles smaller than 5 microns, size of equipment for such separations becomes inordinately large.
Mechanical classifiers are available for size separations from 10 to 325 mesh, or even finer.	Because of theoretical limitations the coarse portion is not completely free from fines, even on homogeneous material.
They operate continuously with little attention and give uniform results.	Because of the pseudoplastic nature of flocculent pulps, a deflocculating agent must often be added for fine separations; this must subsequently be counteracted before clarification.
Operation is flexible, adjustments are simple, and the equipment does not easily get out of order.	
They produce a coarse portion of low moisture content, much freer from undersize than general theory would indicate and, therefore, they make efficient closed-circuit grinding possible.	
Oversize from classifiers is elevated, which makes unnecessary auxiliary elevating equipment beyond the tube-mill scoop, except in large units.	
Machines are available in almost any construction for corrosive solutions; in sizes to handle up to 16,000 tons per day of sand load; and in proportions to handle almost any ratio of coarse to fine in feed.	

Hydraulic Classifiers

ADVANTAGES	DISADVANTAGES
Theory is well understood.	Hydraulic classification requires large quantities of water, especially when free settling.
Hydraulic classifiers of hindered-settling type can produce a series of closely sized products from 6 to 200 mesh on homogeneous feed.	Coarse portion is not drained and fine portion has high moisture content.
On heterogeneous feed, if difference in density is sufficient, hydraulic classifiers can effect considerable concentration of the heavy material.	There is considerable head loss through machines with respect to coarse portion.
Such classifiers require no power except for water used.	Feed must not contain too much very fine material.
	Machines must be built in rather small units to obtain proper distribution of water.

ROTARY MILLS

Ball Mill.—The ball mill, great relatively in diameter in respect to length, is probably the most efficient type of rotary mill in existence. Initially developed for relatively coarse primary grinding, it has held its position there and recently extended its field well into the territory of the tube mill for secondary grinding. The reason seems to be that with the development of efficient classifiers and the system of closed-circuit grinding, it became no longer necessary to finish the grind in a single passage through the mill because the classifier, not the mill, controlled final fineness. Furthermore, economy dictated a short barrel length to reduce inefficient overgrinding and cause a particle to be rejected from the grinding circuit as soon as it had been reduced to the desired size. Ball mills, equipped with extra large feed and discharge trunnions, have efficiently handled circulating loads of 1800 per cent; which means a returned classifier sand tonnage 18 times as great as the new ore feed.

Rod Mills.—The rod mill is a good granulator and frequently is used for a primary grinding. It works well on coarse feed and delivers a fairly uniform product that seldom shows signs of overgrinding, but is limited to low circulating loads.

Tube Mill.—The tube mill, relatively small in diameter and great in length, is well adapted to fine, secondary grinding and finds in the metallurgical field its chief usefulness in cyanidation where the final product may be all through 200 mesh or even only 1 or 2 per cent on 325 mesh. Because of its length and the long time taken for its charge to pass through it, the tube mill tends to overgrind and overslime. While this is not injurious, it is uneconomical and the full advantages of closed-circuit grinding are not realized.

Fine Grinding

Two methods are employed in fine grinding: open circuit, in which the ore is fed to the mill at a rate to produce the finished product required, and closed circuit, in which the ore is discharged unfinished and the finished product separated by a classifier or screen, and the unfinished material returned to the mill. The former method has fallen into disuse. The latter today is practically standard practice.

Open-circuit Grinding.—Open-circuit grinding used to be highly regarded because, when followed by a concentrating device, it permitted mineral to be removed as soon as it became unlocked from the gangue and before the ore had been ground to the ultimate fineness. On the other hand, open-circuit grinding was known to be more costly than closed-circuit grinding, as will be brought out later.

Open-circuit grinding has now generally fallen into disuse at all except the very small properties. For the advantage attending its use from a concentrating standpoint is now secured in a closed-circuit layout in which a concentrating device, such as flotation cell or a corduroy blanket, is placed in the launder leading from the mill to the classifier.

It requires that the material to be ground must remain in the mill until every particle has been reduced to the size specified. To do this, the greater portion remains in the mill long after it has reached the fineness desired, with extra consumption of power and grinding media.

This cannot be justified. Furthermore, the presence of this large amount of overground material tends to defeat the purpose of grinding, as the superfines surround the few remaining coarse particles and prevent effective crushing by the cascading balls. Although careful regulation may limit the maximum size of particle in the product, there is no way to hold the product to a minimum particle size, because, in process of grinding, some of the overground superfine particles are reduced to nearly colloidal size.

Closed-circuit Grinding.—When grinding in closed circuit, the mill is fed at several times its open-circuit rate, the material passes through the mill in a fraction of the usual time and the product entering the classifier contains a relatively small amount of finished material. No attempt is made to finish the reduction in a single pass through the mill, but, rather, every effort is made to remove the material from the system as soon as it reaches the required fineness, thus allowing the balls to work unhampered on the unfinished particles. The fact that capacity increases without corresponding increase in power is attributable to the more rapid elimination of fines, the reduction of uneconomical overgrinding, and the increased amount of coarse material that may be exposed to the cascading balls at one time.

Mill Efficiency and Work Done in Grinding.—The fact that the grinding efficiency of a ball mill or tube mill increases with feed rate has been proved beyond question in the field as well as in the laboratory. The Mines Experimental Station of the University of Minnesota has been particularly active in this work and has published many contributions that throw light upon grinding, some of which are used in this paper.

Screen Analysis—Kick and Rittinger Methods of Computing Work.—In practice the work done in any grinding mill may be measured by the tons of material of the specified size produced in a given period. This may easily be calculated from screen analyses of the feed and the discharge and tonnage determinations. The Kick and Rittinger methods are more refined, in that they take into consideration the work actually done in breaking the substance (Table 1). Rittinger's method is based upon the hypothesis that the work done in crushing is proportional to the square of the diameter of the particle. The units used are called surface units (S.U.). Kick's method is based on the assumption that the work done is inversely proportional to the change in volume of the particles, a function of the cube of their diameters. The units in this case are defined as energy units (E.U.). In Fig. 1 it is shown that the work done increases with increases in the rate of feed, and that a definite relationship, probably expressed by a parabolic equation, exists between feed and work. This relationship holds true whether we gage work by the tons of material of a given size produced, or by applying the Kick or the Rittinger methods.

The 3-ft. ball mill used in certain tests consumed 4 kw. during the entire runs, regardless of the increased amount of work done on the increased feed. Therefore the work done and unit power cost of grinding in this particular case may be expressed as in Table 1.

TABLE 1.—*Ratio of Feed Rate to Power Used*

Feed Rate, Lb. per Hr. of 3/8-in. Limestone	Finished Product in Discharge, Lb. per Hr. of 65-mesh Limestone	Finished Material in Mill Discharge, Per Cent	Rittinger's Units (S.U.)	Kick's Units (E.U.)	Kw-hr. per Ton of −65 Mesh
1,000	600	60	40,000,000	900,000	13.3
2,000	970	48.5	60,000,000	1,500,000	8.25
3,000	1,200	40	90,000,000	2,100,000	6.67
4,000	1,400	35	100,000,000	2,250,000	5.70
5,000	1,650	33	110,000,000	2,350,000	4.85

Effect of High Circulating Loads on Work Done.—As previously stated, the metallurgist, having assured himself from numerous tests that the foregoing relationship exists between feed rate and production of finished product, obtains the advantage of increased capacity by closing the circuit with a classifier. A uniformly fine material leaves the circuit as a classifier overflow, while the classifier rake product returns to the mill to load it properly for efficient grinding.

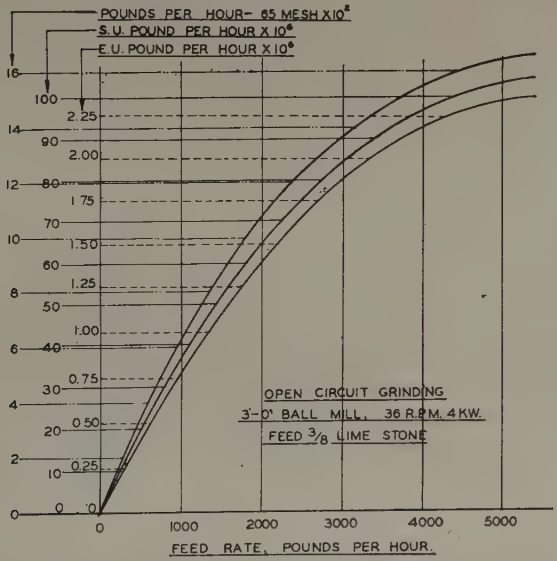

FIG. 1.—CURVES SHOWING THAT WORK DONE ON MATERIAL IN A MILL INCREASES AS FEED RATE INCREASES. (*Courtesy of Minnesota School of Mines.*)

At the Experimental Station cited, many tests approximately of plant scale were conducted to determine the effect of large oversize returns to the mill on the work units produced. The data quoted show that work units increase as the circulating load increases to the point where it amounts to five times the new feed. The curve is still rising at a circulating ratio of 5 to 1, and in practice a circulating ratio of over 18 to 1 has been successfully carried (Fig. 2).

Effect of Circulating Load on Minimum Particle Size.—Closed-circuit grinding reduces the tendency to produce superfines. With, let us say, a circulating load of 500 per cent, the average particle in the final overflow has been passed through the mill five times before reaching the degree of subdivision that allows it to overflow the classifier to subsequent treatment. Accordingly, it goes through a progressive reduction with five times as many opportunities for leaving the circuit as it would have had were it reduced in an open-circuit or single-pass mill. It may be stated, therefore, that proper classification accurately controls the maximum size of the particles in the finished product, whereas high circulating loads and the consequent reduction by stages minimizes the formation of superfines.

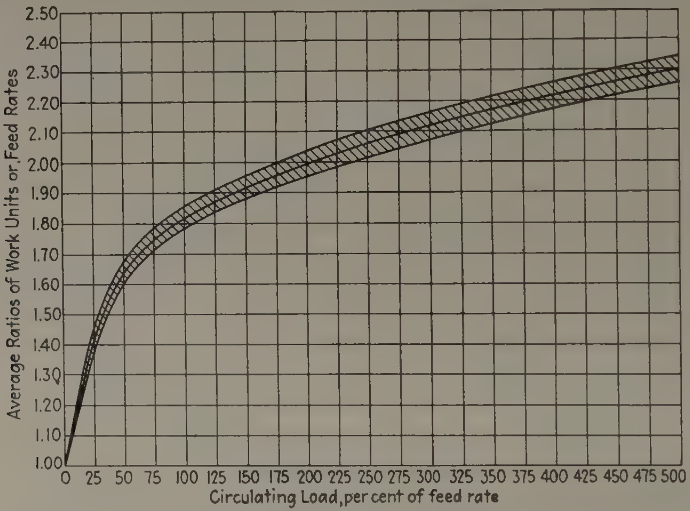

Fig. 2.—Relation between circulating load and work done in ball mill.

Evolution of Multiple-stage Grinding Flow Sheets

The multiple-stage grinding flow sheet of today is an evolution of the last 30 years. Metallurgists now realize that the ratio of the size of the material entering the primary stage of grinding to the size of the material leaving the plant as finished product determines in a general way the number of steps in size reduction. It seems to be fairly well established that any given grinding step suffers a loss in efficiency when a greater reduction of diameter than 4 or 5 to 1 is attempted. Consequently, multiple-stage reduction is practiced in order that each unit in the reduction cycle may operate over the range of its maximum effectiveness.

The first mechanical classifier of 1904, the Dorr, operated in open circuit on Chilean mill product to divide it into two constituents—a slime-free sand for percolation treatment and a sand-free slime for treatment by agitation, decantation and filtration.

The next step, which came with the all-slime method of cyanidation, was the operation of the same classifier in closed circuit with the secondary tube mills, thus controlling accurately the fineness of the pulp and at the same time loading the mill with returned sands to a more efficient load.

With the introduction of ball mills and rod mills to replace stamps for primary grinding, the classifier again was used to close the circuit and to act as a convenient means of apportioning the work.

Then came the interposition of a bowl classifier between stages. This classifier operated in open circuit, receiving as its feed the overflow from the primary circuit; discharging a clean sand product to the secondary circuit and overflowing material of finished size to treatment.

Recently, some operators have found it advantageous to add a third stage of classification and grinding, in order that all of the feed to concentration or extraction treatment may be finished in a single large bowl classifier in circuit with a mill for regrinding stray oversize and the heavy and valuable mineral that becomes concentrated readily in the sand product of a bowl classifier.

Another step of comparatively recent origin is the addition of a preliminary desliming classifier, directly ahead of the primary, closed-circuit grinding unit and after the last stage of crushing. Thus, primary slime is removed at the outset and sent direct to treatment or to some succeeding stage in the reduction cycle.

It should not be construed from the foregoing that all of these steps are to be recommended. In certain cases the removal of primary slime in either the preliminary or intermediate classifier is actually detrimental to smooth classifier operation in the primary and secondary circuits. On the other hand, there can be no question about closing the primary and secondary circuits, also the tertiary circuit if there is one.

Typical Flow Sheets

The flow sheets in Figs. 3, 4 and 5 show how the grinding sections of a number of copper concentrators were set up in 1929, the last year of peak production, when a cost study was made of nine plants and presented in a paper by J. V. N. Dorr and A. D. Marriott.[1]

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[1] J. V. N. Dorr and A. D. Marriott: Importance of Classification in Fine Grinding. *Trans.* A.I.M.E. (1930) **87**, 109.

Fig. 3 shows the flow sheet of United Verde Copper Co., Jerome, Arizona. This is a straight two-stage, closed-circuit system, similar to that of Braden Copper Co. in Chile and Wright-Hargreaves, Sylvanite and others in Canada. United Verde at that time ground to 80 per cent minus 200 mesh and carried a 3 to 1 primary and a 5 to 1 secondary circulating load. Wright-Hargreaves and Sylvanite grind to only 1 or 2 per cent on 200 mesh and carry 7 to 10 primary circulating loads with extra heavy, large classifiers.

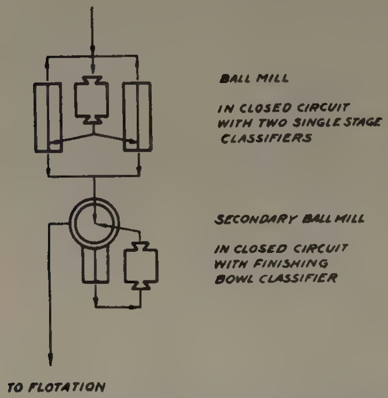

BALL MILL
IN CLOSED CIRCUIT WITH TWO SINGLE STAGE CLASSIFIERS
SECONDARY BALL MILL
IN CLOSED CIRCUIT WITH FINISHING BOWL CLASSIFIER
TO FLOTATION

Fig. 3.—Grinding flow sheet, United Verde Copper Co. Two sections total.

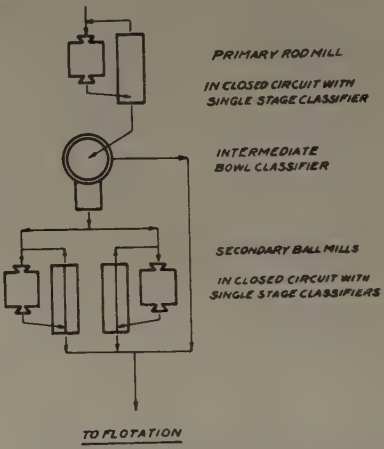

PRIMARY ROD MILL
IN CLOSED CIRCUIT WITH SINGLE STAGE CLASSIFIER
INTERMEDIATE BOWL CLASSIFIER
SECONDARY BALL MILLS
IN CLOSED CIRCUIT WITH SINGLE STAGE CLASSIFIERS
TO FLOTATION

Fig. 4.—Grinding flow sheet, Cananea Consolidated Copper Co. Three sections total.

The flow sheet in Fig. 4 is that of the Cananea Consolidated Copper Co., Sonora, Mexico, a typical two-stage, closed-circuit set-up with an intermediate open-circuit bowl classifier. A similar scheme was found in 1929 also at the Nevada Consolidated Copper Co., McGill, Nev., and the Magna mill of the Utah Copper Co. Cananea ground to 73.1 per cent, Nevada to 73.4 per cent, and Magna to 67.11 per cent through 200 mesh.

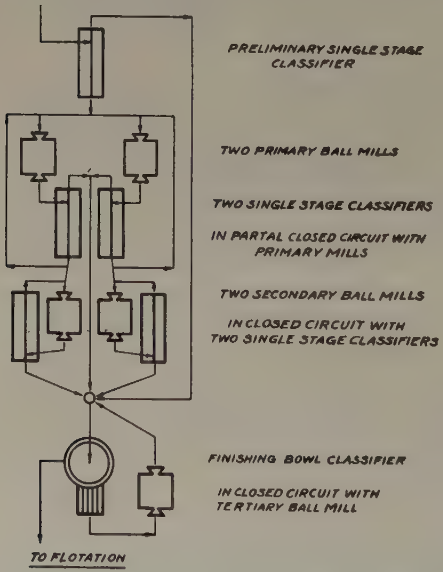

PRELIMINARY SINGLE STAGE CLASSIFIER
TWO PRIMARY BALL MILLS
TWO SINGLE STAGE CLASSIFIERS
IN PARTAL CLOSED CIRCUIT WITH PRIMARY MILLS
TWO SECONDARY BALL MILLS
IN CLOSED CIRCUIT WITH TWO SINGLE STAGE CLASSIFIERS
FINISHING BOWL CLASSIFIER
IN CLOSED CIRCUIT WITH TERTIARY BALL MILL
TO FLOTATION

Fig. 5.—Grinding flow sheet, Miami Copper Co. Six sections total.

The flow sheet in Fig. 5 is that of Miami Copper Co., Miami, Arizona, a triple-stage layout with a preliminary classifier at the head end for removing primary slime at the start. Granby Consolidated Mining, Smelting and Power Co., Allenby, B.C., had a somewhat different three-stage plan without the preliminary classifier, but with an intermediate bowl classifier between stages. Miami ground to 53.8 per cent and Granby to 62 per cent through 200 mesh.

An investigation of the power consumed for fine grinding and classification at the nine plants studied revealed the figures given in Table 2. The indices of comparison used—kilowatt-hours per ton 100 mesh and kilowatt-hours per ton 200 mesh—were derived in each case by multiplying the rated tonnage by the difference between the percentages of plus 100-mesh (or plus 200-mesh) material in the feed and in the finished product, both percentages being expressed as decimal fractions.

TABLE 2.—*Unit Power Consumption for Fine Grinding*

Plant	Kw-hr. per Ton 100-mesh	Kw-hr. per Ton 200-mesh
1	7.90	10.80
2	8.53	9.73
3	8.95	11.66
4	9.20	11.69
5	10.10	13.10
6	10.61	14.56
7	10.74	14.80
8	11.93	15.31
Average..............	9.74	12.70

IMPORTANCE OF CLASSIFIER IN CIRCUIT

While it is true that the mill is the grinder, it is equally true that the classifier controls the system. Not alone does it control the fineness of the product, for probably its most important function is to load the mill with circulated sands so that the utmost effective work is obtained from the cascading grinding media with minimum destruction of the media and liners themselves.

The following statements have been proved in practice and are supported by many data in addition to those quoted. These data necessarily have been limited to information obtained from contact with the type of classifier most familiar to the authors; i.e., the Dorr classifier and the Dorr bowl classifier.

Examples of Closed-circuit Grinding

Closed Circuit Increases Mill Capacity.—When grinding a substance so that all of it shall pass a screen of a given size, the capacity of the mill may be increased by closed-circuiting it with a classifier, as shown by the examples in Table 3.

TABLE 3.—*Increase in Mill Capacity by Closed Circuiting*

Item	Examples		
	1[a]	2 (Wright-Hargreaves)	3 (Morenci)
Size of mill, ft	6 by 20		
Feed, mesh	−6		
Product, mesh	8% + 100	−20	−65
Capacity, tons per day			
Open circuit	144	190	89
Closed circuit	240	275	174
Increase	96	85	85
Increase, per cent	66	44.7	95.5

[a] Quoted from A. F. Taggart: Handbook of Ore Dressing.

Classifier Determines Capacity.—The capacity of the closed circuit is frequently, if not always, determined by the classifier, not by the mill; in other words, additional classifiers increase the capacity of the circuit without any increase in size of the mill. For example: (1) The Tough-Oakes Gold Mines, Ltd., Kirkland Lake, Ontario, by using two classifiers in closed circuit with a given mill, made the capacity of the circuit 28 per cent greater than the capacity of the mill in closed circuit with a single classifier. (2) The Consolidated Mining and Smelting Co. of

TABLE 4.—*Reduction in Power by Closed Circuiting*

Item	Examples[a]	
	2. Lucky Tiger	3
Size of mill, ft	5 by 14	6 by 20
Feed, mesh	11 to 20% + 20	−6
Product, mesh	−100	10% + 100
Open circuit:		
Capacity, tons per day	22	144
Power used, kw	47 (hp.)	75
Minus 100 mesh, tons per hp-hr	0.016	
Plus 100 mesh, tons per hp-hr		0.0565
Closed circuit:		
Capacity	37	240
Power used, kw	47 (hp.)	65
Minus 100 mesh, tons per hp-hr	0.055	
Plus 100 mesh, tons per hp-hr		0.1087
Unit power cost reduced, per cent	71	48

[a] From A. F. Taggart: Handbook of Ore Dressing.

Canada, Kimberly, B.C., by operation similar to that of the Tough-Oakes, increased the capacity 35 per cent.

Closed Circuiting Reduces Unit Power Cost.—The power required to drive a given mill remains practically constant, regardless of tonnage fed, and, accordingly, increased capacity, due to closed circuiting, results in diminished power costs per ton finished product.

At Lake Shore Mines, Kirkland Lake, Ontario, closed circuiting increased the capacity 44.70 per cent and power consumption was reduced 10 per cent, due probably to better balance with heavy feed. The reduction in power per ton of finished product was 37 per cent. (For other examples see Table 4.)

Closed Circuiting Reduces Wear on Liners and Balls.—Through a better loading of the mill with coarse classifier oversize, the abrasion of metal liners and the consumption of grinding media are greatly reduced, as shown by the following examples:

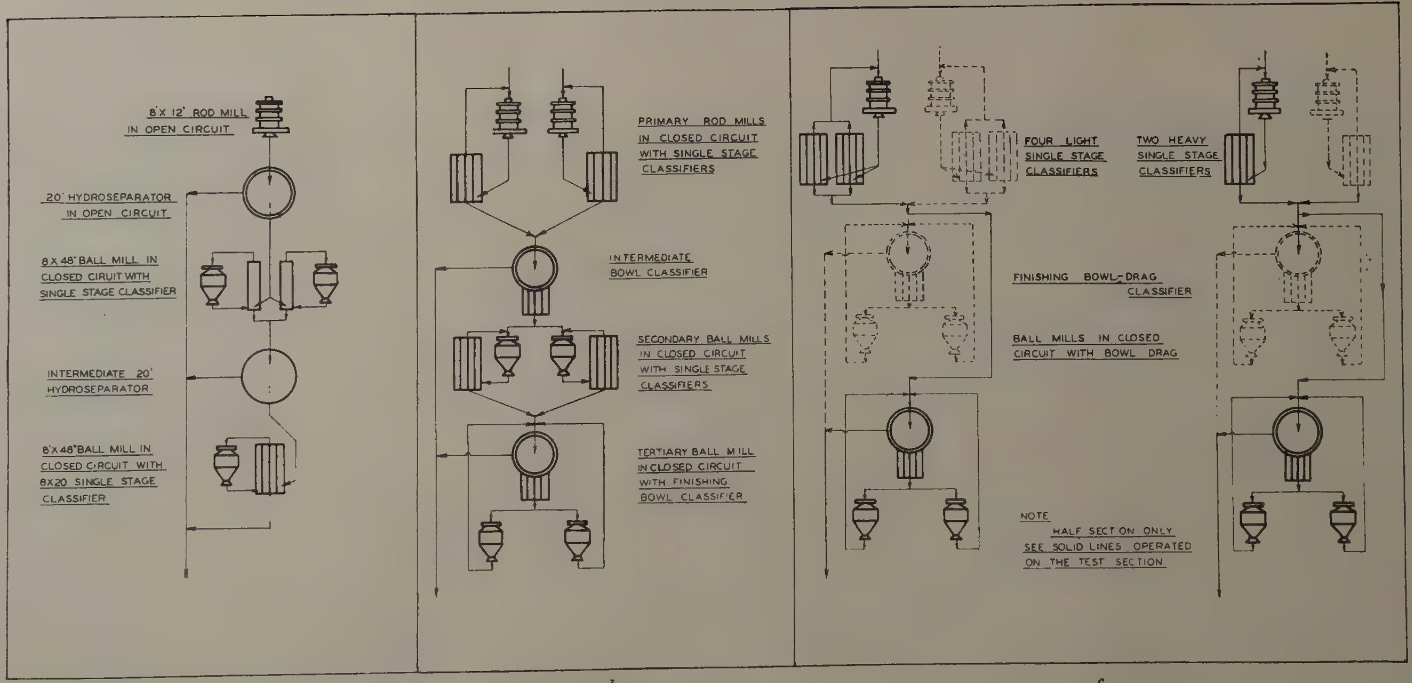
1. The Lake Shore Mines had a steel consumption of 6.5 lb. per ton of finished product in the open circuit and 3.2 lb. per ton in the closed circuit, a reduction of 51 per cent.
2. The Chino Copper Co., with one mill and one classifier in closed circuit, had new feed of 150 tons per day. The ball and liner wear was 3.2 lb. per ton of finished product. With one mill and six classifiers in closed circuit, having new feed of 240 tons per day, the ball and liner wear was 1.5 lb. per ton of finished product. The reduction in steel loss was 53 per cent.

Summing up, it may be stated that in the field of metallurgy, both theory and practice agree on the desirability of close-circuiting wet-grinding mills with classifiers, because closed-circuit operation increases capacity, reduces unit power cost of grinding, cuts down the wear on liners and grinding media, and in general permits the mill to be loaded to its maximum output of material of a specified size. These advantages have the cumulative effect of reducing the unit cost of grinding, controlling accurately the size of the maximum particle in the finished product, and limiting the tendency to overgrind a large portion to semicolloidal size.

RECENT TREND TO HIGH CIRCULATING LOADS

The advantages of high circulating loads, as brought out in the foregoing, have been known for years. In practice, however, circulating loads were not generally carried to the optimum point until comparatively recently, largely because a number of relatively light classifiers had to be used to secure the necessary sand-raking capacity.

With the introduction two years ago of heavier classifiers, having sand-raking capacities three to four times as large as the former light



a. Initial Flow Sheet (not sectionalized)
Three-stage grinding with intermediate hydroseparators between stages.

b. Second Flow Sheet
Three-stage closed circuit grinding with one intermediate bowl classifier.

c. Third and Final Flow Sheet

A. Two-stage closed circuit grinding with two light duty classifiers in circuit with each primary mill.

B. Two-stage closed circuit grinding with a single heavy duty FX classifier in circuit with each primary mill.

	a.	b.	c.
Ore character:			
"Soft," per cent	86	40	40
"Hard," per cent	14	60	60
Tons original feed per primary mill	1238	1100	1350
Tons 100-mesh produced:			
Per primary mill	314	357	504
Per secondary or tertiary mill	176	233	270
Number of secondary and tertiary mills per primary mill	3.12	2.0	2.0
Flotation feed, per cent:			
+100 mesh	16.4	4.3	3.1
−200 mesh	67.2	75.3	78.2
Solids	18.5	18.0	18.6

FIG. 6.—PROGRESSIVE CHANGES IN GRINDING AND CLASSIFICATION IN ONE MILL.

classifiers of the same size, the deterrent to high circulating loads in commercial milling was removed. For, with these high-capacity classifiers sand-raking capacity could easily be trebled or quadrupled without making further demands on the space reserved for classification equipment.

These heavy classifiers weigh twice as much as their predecessors and under certain conditions rake as much as 1000 tons of sand per day per foot of rake width. Their use has led to the equipping of the mills with extra large trunnions and feed scoops and to the use of several different schemes for circulating the pulp and sand in the circuit, including an ingenious spiral scoop elevator and pipe launder for elevating mill discharge to the classifier, thus permitting gravity return of the sands to the mill.

At least four plants are now holding their circulating loads in the 8 to 10 to 1 range commercially day in and day out. Two of these have handled circulating loads of 13 to 14 to 1 for a day or so at a time but have not as yet stabilized operations permanently at the higher levels. The classifiers used are generally 8, 12 or 16 ft. wide and there is no mechanical reason why they cannot be built 20 ft. wide or wider.

This present trend to higher circulating loads is brought out in flow sheets with corresponding tables of operating data in Fig. 6. There it is shown how one representative concentrator recently progressively modernized its grinding sections. By installing adequate classification capacity and simplifying its flow sheet the operator: (1) increased the minus 100-mesh output of its entire grinding plant 34.7 per cent; (2) eliminated the need and cost of 2.5 secondary and tertiary mills per one primary mill; (3) produced a 3 per cent plus 100-mesh flotation heading with about a similar percentage of solids as the initial flow sheet making a 10 to 15 per cent plus 100-mesh heading; (4) reduced the tailing loss one-third; and (5) increased the copper recovery 4 to 5 per cent.

Savings 1 and 2 were undoubtedly due to increasing classification capacity from 3¾ ft. of rake width per mill initially to 9⅓ ft. per mill finally, and closing the primary circuit with a heavy-duty classifier that could properly load the mill with a 9 to 1 circulating load.

Saving 3 was a result of adequate bowl area in the secondary circuit, permitting a much finer separation without injuriously affecting the percentage of solids.

Savings 4 and 5 are a natural result of the finer flotation heading produced by the bowl classifier, plus the fact that the concentration of heavy valuable mineral in the rake product assured its being ground selectively to a finer degree and thus made more amenable to flotation concentration.

An evaluation of the savings made showed a return of the investment in equipment, auxiliaries, and installation charges in about a year and the change-over has been going forward for some time, section by section, and will continue until the whole mill is modernized. At present, 10 or 12 to 1 primary circulating loads and 6 to 1 secondary circulating loads are normal practice at this concentrator.

MECHANICAL REQUIREMENTS OF AN EFFICIENT FINE-GRINDING PLANT

The design of the flow sheet best adapted to a given ore and given set of conditions is a matter of specific information on the problem in hand, tempered by broad knowledge and experience in milling and classification. Each case is separate and unique, and no set of rules can be laid down for its solution. Excepting the most unusual cases, it can be said that at least two stages of fine grinding will be needed to reduce crushed ore to from 65 to 200 mesh and that both stages of grinding should be close-circuited with classifiers. In certain cases, especially where very fine grinding is required, a tertiary stage may prove desirable. Local conditions, particularly the relative primary slime content of the ore, will show whether an intermediate bowl classifier will prove helpful or whether a preliminary classifier, ahead of the first stage of grinding, may be desirable.

On the mechanical set-up, however, some basic rules may be stated, which are generally accepted by experienced operators:

1. Rotary mills should be relatively great in diameter and short in length and provided with extra large feed and discharge trunnions to accommodate circulating sand loads at least 10 times the new feed.
2. Spiral feed scoops should be of extra heavy construction, armed with abrasive resisting tips and renewable internal scoop liners.
3. Provision should be made for measuring the loss of grinding media and making up deficiencies at frequent intervals.
4. Wattmeters should be installed to serve as guide to the correct loading of the mill.
5. The classifier should be of rugged simple construction, large enough to carry an adequate circulating load with ease.
6. Suitable meters should be installed to show power consumption, which broadly is a function of the sand tonnage handled.
7. A constant head tank for dilution water should be available, so that the rate of addition of water may be regulated to a nicety and then held constant. The dilution of the overflow should be checked constantly, either manually or by some type of hydrometer connected with the tank.
8. Classifiers may generally be installed at such an elevation and at such a distance from the rotary mill that an all-gravity closed circuit is secured; i.e., mill discharge flows by gravity to the feed launder of the classifier and classifier sands similarly gravitate to the feed box of the mill. Where this proves impossible, it is preferable to locate the classifier at such an elevation that sands return to the mill by gravity, providing auxiliary apparatus for raising the more dilute and less abrasive mill

discharge to the classifier feed launder. For the latter purpose a rotary pipe launder and spiral pickup scoop has been used to advantage in several cases.

Acknowledgment

The authors have made use of certain material published by others in the preparation of this paper, notably "Handbook of Ore Dressing" by Taggart and "Classification" by Hubler and Martin [*Canadian Min. Jnl.* (July, 1934)]; also former publications of their own, including "Half a Century of Cyanidation" by Anable [*Canadian Min. Jnl.* (Feb. 1929)]; "The Importance of Classification in Fine Grinding" by Dorr and Marriott [*Trans.* A.I.M.E. (1930) **87**, 109]; and "Closed-Circuit Fine Grinding" by Anable [*Rock Products* (Jan. 5, 1929)].

Acknowledgment is also gratefully made to members of the profession and to members of the senior author's staff who have assisted in the collection of material and the reviewing of the manuscript.

Colloidal Chemistry of Pulp Thickening

By Elliott J. Roberts*

In spite of the fact that the percentage of true colloids in an ore grind is very small,[1] these pulps exhibit many of the properties of true colloidal suspensions. A quartz particle, appreciably finer than 10 microns in diameter, has a settling rate so low that at a dilution of 5:1 or over, the capacity of a settling tank to settle such particles would be uneconomical if the individual particle rate prevailed. However, by grouping the finer particles into aggregates or flocs, the settling rate can be increased to an economical value.

The colloidal chemistry of pulp thickening is thus chiefly concerned with particles having diameters of between 1 and 10 microns, although particles as coarse as 40 microns in diameter are more or less involved. These particles must be associated in such a manner that they settle at an economical rate and give a clear, supernatant liquid or overflow.

The treatment of the subject may be divided into three phases, each phase having a more or less different colloidal aspect. The first phase is the problem of getting the pulp or suspension into the proper condition of flocculence; the second is the behavior of the flocculent pulp in its initial and intermediate stages of settling; the third is the behavior of the flocs in the final period of settling or thickening stage.

Let us look first into the methods and mechanism of obtaining the flocs and then consider their behavior and properties.

Flocculation

Colloids are merely ordinary substances reduced to a very small particle size, the upper limit being placed by definition at 1 micron (0.001 mm.) diameter by the soil chemists and at from 0.2 to 0.1 micron by various other colloid chemists. The lower limit is reached only when the particles approach molecular dimensions. The term "colloid" should not necessarily imply lack of crystallinity. Particles as small as 0.01 to 0.02 micron give perfect X-ray patterns, while with 0.005 micron particles the bands on the powder photograph are merely broadened and diffused.[2]

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[1] A. J. Weinig: Colo. School of Mines *Quarterly* (1933) **28**, No. 3, chap. 9.

[2] Clark: Colloid Symposium Monograph, **4**, 145 et seq.

Colloids are divided into two major classes; sols, or suspensoids and gels, or emulsoids. Sols are distinguished from gels by the fact that they are very sensitive to the action of electrolytes, while gels are not. In pulp thickening we are concerned only with the sols.

When sols are suspended in water under proper conditions, they disperse and will not settle under the influence of gravity. The most generally accepted theory explaining the behavior of such a colloid dispersion is the electric double-layer conception of Helmholtz. The colloid particle is conceived to have, either attached to itself or as a part of itself, ions that bear an electric charge. These ions form the inner wall of the double layer. In the water surrounding the particles are ions of the opposite charge, equal in total charge to the total charge of the particle. These ions form the outer wall of the double layer and together the two walls constitute a spherical condenser. At any appreciable distance from the particle, the net effective charge of the particle is zero. However, as two such particles approach each other at close range there is a repulsion, due to the fact that particles behave as condensers with the outer layer a conductor. When the effective potential is above a certain minimum value the colloid is stable; the impacts caused by Brownian movement are not sufficient to cause coalescence of the particles. Helmholtz pictured the outer wall as a discrete layer, electrically charged, at a distance of only one molecular diameter from the inner electrical layer. Modern theories of solution have necessitated a modification to the extent that the outer layer is now considered to be diffuse and not concentrated at one molecular diameter from the other layer. Thus the double layer of ions keeps the particles from uniting and the Brownian movement or bombardment by the water molecules keeps them from settling under the influence of gravity.

The ions forming the inner layer are commonly referred to as adsorbed ions. In the majority of cases these bear a negative charge, although with certain substances they are commonly positive. There are many theories as to why the ions are adsorbed and some hold that with certain substances the charge is due to ionization; e.g., quartz becomes negatively charged owing to ionization of silicic acid formed on its surface. Many substances may adsorb either negative or positive ions, depending on the composition of the electrolyte in which they are suspended. However, they must adsorb some ion, and electrolytes that can furnish suitable ions are called dispersing agents. Thus ferric oxide is dispersed by ferric chloride because the oxide adsorbs ferric ion quite strongly. Clays are dispersed by alkalis, presumably because of the adsorption of hydroxyl ion. Sodium silicate is a very catholic dispersing agent, being effective in many instances. This is attributed to the combined effect of the hydroxyl ions, which are formed by hydrolysis and generally are adsorbed quite strongly, and the negatively charged colloidal SiO_2

present, which also is apparently strongly adsorbed. The salts of a great number of organic substances are also good dispersing agents.

It was stated earlier that one of the characteristics of sols is that they are very sensitive to the action of electrolytes. If an electrolyte is added to a sol in sufficient amount, the stability of the sol is destroyed and flocculation ensues. The generally accepted picture of the mechanism of flocculation is that the particles adsorb ions of the opposite charge; these new charges counteract the effect of the stabilizing charges and the electric double layer is destroyed, with the result that actual impacts between the particles are now possible and the sol flocculates. The true mechanism may be somewhat different from this but the newer ideas are in a state of flux; this description affords a working picture.

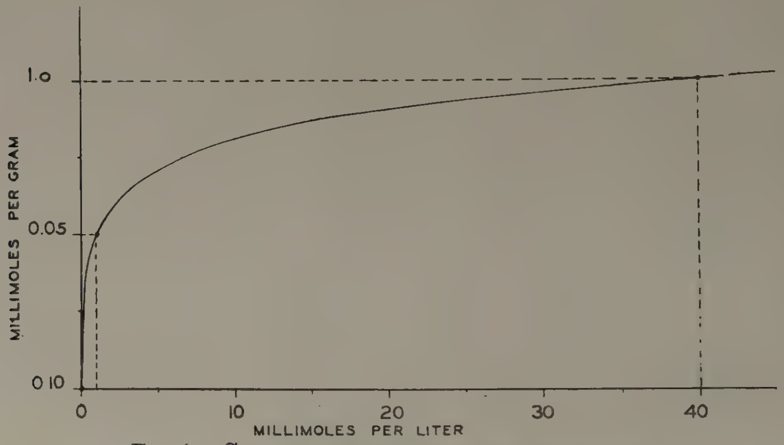

FIG. 1.—CURVE OF TYPICAL ADSORPTION ISOTHERM.

Although there are many exceptions, the sol is generally more sensitive to polyvalent ions whose electric charge is opposite to that carried by the colloid. Thus a divalent positive ion such as Ca^{++} will flocculate a negative sol such as clay or silica at a much lower concentration than a univalent ion such as Na^+. In some cases only $\frac{1}{40}$ as many moles of the divalent ion are required. Trivalent ions are still more effective in general. There are, however, many exceptions to the above; hydrogen, organic and heavy metal ions are most notably out of line, their action being much more powerful than one would expect from their valence. Freundlich[3] explained the increased effectiveness of divalent ions on the basis of the adsorption isotherm. In Fig. 1, the curve represents a typical adsorption isotherm in which millimoles of ion adsorbed per gram of colloid is plotted as ordinate and the concentration of the ion in the solution is plotted as abscissa. Suppose, for example, it takes 0.1 milliequivalents of adsorbed ion to flocculate the sol. Then it will take a concentration of 40 millimoles of monovalent ion per liter to flocculate it according to the curve given. For a divalent ion, only 0.05 millimoles is required to furnish the 0.10 milliequivalent and since the curve is convex upward, the concentration of ion required is much less than ½ of 40; on the curve shown it is 1.0 millimole per liter. This explanation involves two assumptions, which Freundlich supported with much evidence: (1) that a constant number of charges is required to flocculate the sol independent of the valence or character of the flocculating ion and (2) that all normal ions, whether monovalent, divalent or trivalent, are adsorbed to the same extent. Kruyt[4] gives a fuller discussion of this subject.

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[3] Freundlich: *Ztsch. physik. Chem.* (1910) **73,** 385.

As mentioned before, the amount of truly colloidal material in an ore grind is very small. However, there may be an appreciable amount of minus 5-micron material.[5] There is no apparent reason why the conceptions of formation and discharge of the double layer applied to the truly colloidal sizes should not be extended to coarser sizes. The only question is whether the material will have enough Brownian movement to hit other particles and stick. Gaudin has made calculations that indicate that, given an appreciable depth, particles as coarse as 5 microns may flocculate under proper conditions. Thus it is possible that all particles smaller than about 5 microns will accumulate into flocs when the pulp is dosed with flocculating agents in the correct amount. If particles in the range of 1 to 5 microns do not aggregate, the tendency of an ore pulp to flocculate must be attributed to the flocculation of the finer material with entrainment of the coarser material.

The particles of a dispersed ore grind possibly may bear either a positive or negative charge, depending on the constitution of the ore and the electrolytes present in the suspending medium. The majority undoubtedly are negative, however, because silica and clay particles normally take a negative charge. Calcium compounds are known to be good flocculating agents for clays. It has been stated previously that hydroxyl ions tend to stabilize clays, so that it might be thought that calcium hydroxide would stabilize rather than flocculate clay. However, the effect of the divalent calcium ion is so much more powerful than the other that it prevails, and lime is actually a good flocculating agent for clays and ore pulps. Theoretically, a calcium salt with a univalent, normal ion such as chloride or nitrate would be better mole for mole, and some experimental results support this view. On the other hand, a mole of anhydrous calcium chloride weighs exactly twice as much as a mole of pure calcium oxide. To be more efficient on a weight basis, therefore, calcium chloride would have to be more than twice as good as lime. Another consideration, of course, is cost and availability, which generally are also in favor of the lime.

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[4] H. R. Kruyt: Colloids, Ed. 2. 1930.

[5] A. M. Gaudin: Flotation, 142. New York, 1932. McGraw-Hill Book Co.

Another reason which may explain why lime is universally so good in flocculating ore pulps is that lime also flocculates iron oxide sol very effectively because of the very intense adsorption of the abnormal hydroxyl ion by the positive iron oxide sol. Thus, if the particles of the ore pulp carried a positive charge and were at all like iron oxide, lime would be as effective on them as on a negative pulp.

As a negative colloid, an ore pulp should be flocculated by acids (hydrogen ion) but less efficiently than by calcium salts, although one cannot always be sure whether a divalent ion will be more effective than the abnormal hydrogen ion. Other normal divalent ions, such as magnesium, barium, strontium and zinc, should behave in much the same way as calcium. The heavy metal ions, lead and copper, should be more effective. The trivalent ions, aluminum and iron, should surpass all except perhaps the lead and copper ions. It should again be emphasized that all these relations are on a molecular basis, and, as pointed out in regard to lime, the situation may be reversed on a weight basis.

If the ore is a positive colloid, bases (hydroxyl ion) should be effective in promoting flocculation. Divalent ions such as sulfates, trivalent ions such as phosphates and tetravalent ions such as ferrocyanide should be increasingly effective in the order given.

In judging the effect of flocculating agents, it must be remembered that a pulp may be thoroughly flocculated and still have a very slow settling rate. About the best criterion of good flocculation is the clarity of the supernatant liquor. A well flocculated pulp should give crystal clear supernatant solution. If the pulp is excessively thin and does not settle with a line, isolated small flocs may remain in the upper part of the solution long after the majority of the solids have settled, but the liquid itself should not be at all smoky or hazy. In other words, it is one thing to flocculate a pulp but quite another to give it a good settling rate. Very little is known about the subject of shrinking flocs or of forming compact flocs, especially in concentrated pulps.

The effect of temperature on the settling rate of pulps is varied. Some investigators[6] have shown that the increase in settling rate due to the increase in temperature was exactly in proportion to the decrease in viscosity of the water. This would be expected if the pulp was thoroughly flocculated to begin with, provided further that no shrinking of the flocs took place as the temperature was raised. On the other hand, some pulps are much more sensitive to the effect of temperature. A change of a few degrees in temperature changes the settling rate far more than can be explained by the change in viscosity of the solution. Such behavior must be attributed either to an increase in the state of flocculation or to a shrinkage of the already formed flocs. In the former, the pulp must be incompletely flocculated at the lower temperature.

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[6] Nichols: *Min. & Sci. Pr.* (1908) **97**, 54.
Free: *Eng. & Min. Jnl.* (1916) **101**, 243, 429, 509, 681.

General Behavior of Pulps

The behavior of the flocculent pulp in its initial and intermediate stages of settling brings us to the second phase under consideration. No better method of arriving at a clear conception of the visible aspects of pulp settlement is apparent than to reproduce Coe and Clevenger's diagram[7] showing the "various stages of slime-settling." (Fig. 2.)

In Fig. 2, cylinder *E* represents the pulp sample after thorough mixing. Cylinder *F* represents the pulp after some slight settlement has taken place. Zone *A*, of clear solution, has formed; just below it lies zone *B*, which is pulp of the consistency of the feed minus any coarse material that has segregated. Below zone *B* is a "transition zone," *C*, which may be vanishingly thin in some pulps and very important in others. It is of varying concentration from top to bottom. Zones *B* and *C* are commonly designated as free settling, as distinguished from zone *D*, which is the compression zone. Zone *D* consists of flocs in rather intimate contact; somewhat like a pile of sponges. Cylinders *G* and *H* are similar to *F*. The relative thicknesses of zones *B* and *C* are not necessarily the same as shown in the diagram; zone *C* may be of infinitesimal thickness or, in a different type of pulp, it may have filled the entire space between zones *A* and *D*, with the consequent elimination of zone *B*. What is known as the critical point is pictured in cylinder *I*. Zones *B* or/and *C* have just disappeared; i.e., zones *A* and *D* have just made contact. Cylinder *J* shows the pulp in the final stage of settling, or in what is known as the final dilution. The pulp is as thick as it will become under the given conditions.

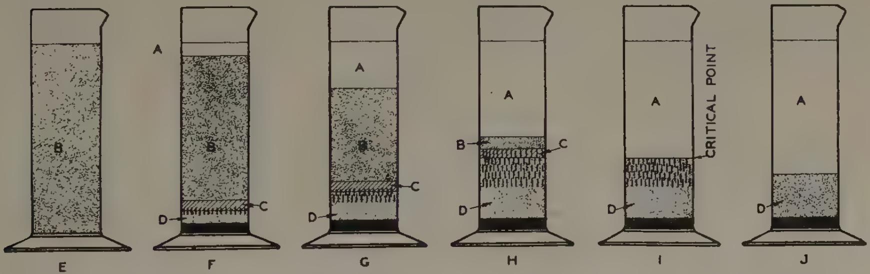

Fig. 2.—Six phases of sedimentation illustrating intermittent thickening (*Coe and Clevenger: Trans. A.I.M.E., vol.* 55).

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[7] H. S. Coe and G. H. Clevenger: *Trans.* A.I.M.E. (1916) **55**, 356.

In picturing the various stages of settlement, as in Fig. 2, it is assumed that the original, well mixed pulp has a certain degree of flocculence and has a dilution (ratio of weight of water to weight of solids) lying between two fairly well defined limits. One of these limits is the dilution of the critical point and the other is the dilution at which the pulp refuses to settle with a sharp line to leave zone *A* clear. Both limits will vary with the degree of flocculence.

In a random pulp sample, even though it be consistent with the above assumptions, not all the zones and phenomena are immediately apparent or visible. By assumption, zone *A* will be sharply defined, as will, of course, the upper limit of zone *B*. There may be no other visible hiatus to the bottom of the cylinder. Often, however, zone *D* is more or less sharply defined by the presence of vertical channels. Furthermore, when the critical point is reached, zone *D* is sharply defined. On the other hand, the critical point is not always readily located, as explained below, in discussion of the third and final phase of pulp thickening.

BEHAVIOR IN THE FREE-SETTLING ZONE

Two types of line settlement were noted by Coe and Clevenger. In type 1 the upper surface of the solids descends at a constant rate until the critical point is reached, where the rate of settlement falls off markedly. The concentration of the settling pulp remains constant and equal to that of the feed dilution minus any segregated material. The behavior is similar in some respects to the isothermal compression of a saturated vapor. In type 2, the settling rate decreases constantly from the start and, although the slowing up is more pronounced at the critical point, sometimes this point is not clearly defined. In this case the concentration of the settling pulp is not constant from top to bottom and is not equal to that of the feed dilution minus segregated material at any point once settling is well started. The analogy here is the adiabatic compression of a saturated vapor.

Coe and Clevenger explained the difference between the two types on the basis of the amount of water carried by the flocs. Another way of looking at it is that in type 1 the rigidity of the floc arrangement pattern is small in comparison to the resistance to flow through the pulp, while in type 2 the structure is more rigid. Thus in type 1, as long as there is a relatively large volume of water flowing through the floc structure, the flocs are held to their original spacing. As soon as they meet an appreciable solid resistance the spacing collapses and the flocs come into close contact. In type 2 the structure would have more strength and elasticity and the flocs would not come into close contact immediately but tend rather to build, in continuously diminishing concentration, from the bottom up.

Just exactly what determines whether a pulp will settle according to type 1 or type 2 has not been explained. Whether it is a function of the amount or character of the truly colloidal material present, the degree of flocculation, the size distribution of the plus 1-micron material, or some other variable would form a very interesting, although probably difficult subject for investigation.

Compression of a Pulp

After a pulp reaches the critical point, further settling results from compression of the pulp; i.e., liquid is forced out from within and between the flocs. Generally vertical channels are visible along the edge of the pulp up which the water rises; the presence of the channels is also indicated by the presence of tiny mounds all over the surface of the compressing pulp which presumably have been thrown up by the ascending liquid.

Such compression or thickening of a pulp will continue at an asymptotically decreasing rate until no perceptible settling occurs over a period of an hour or two. If the pulp is stirred the limiting dilution will be lower than in the unstirred case. This stirred limiting dilution is ordinarily called final dilution.

Not infrequently, if allowed sufficient time, pulps will thicken to a mass having such a high elastic limit or yield point that it is not practicable to pump them. The remedy, of course, is to withdraw the pulp at a higher dilution, which is pumpable, and not give it the detention required for the extreme condition.

With pulps whose critical points occur at rather high dilutions and which settle slowly, the thickening process often may be very much hastened by stirring with a picket fence arrangement, as in the gluten-type thickener. The stakes of the picket fence allow the ready formation of vertical channels, which aid in the elimination of water.

In general, a pulp in the compression zone exhibits a rate of concentration that is a function of the concentration and not position or volume. That is, a unit volume of the pulp at a given concentration will eliminate the same amount of water per minute no matter what its vertical position in the column of pulp. This is deduced from the fact that, with certain limitations, the settling rate of a given compression pulp doubles if a double depth of pulp is used. The limitations indicated by Coe and Clevenger are: (1) Time must be allowed for the development of channels, and (2) the rate cannot be increased beyond that characteristic of the pulp at a dilution just above that of compression.

The practical result of this behavior is that a certain volume for taking a pulp to final dilution is required, which can be supplied by choosing a depth of thickener that in conjunction with the area requirement imposed by the free-settling rates will give the required volume.

If headroom or other measurements indicate that the depth is too great, the volume can be supplied by increasing the area of the machine.

Special Behaviors

Certain variations occur in the behavior of a pulp if it is not too dilute or too dispersed. In these cases zone A is not clear. Sometimes a poor line forms after a time and sometimes this line may become very sharp, leaving a layer of clear liquor between the cloudy top layer and the subsiding line. On occasion a fair line may be observed almost from the start, with the supernatant liquid very turbid. Other samples may show no line except that of a compression layer building up from the bottom at first and then finally subsiding. Completely dispersed pulps show a line rising from the bottom, which does not subside. This layer is very compact from the start and is entirely different from a compression pulp even at final dilution.

Zone Tests

If a normal pulp—one sufficiently concentrated and flocculated—is well mixed at a given dilution and allowed to settle, a certain settling rate will be observed, which may be constant until the critical point is reached if the pulp is of type 1. If the solids are repulped at a lower dilution, a lower settling rate will be observed, which also may stay constant to the critical point. Thus it is noted that the rate depends on the *initial* dilution and not the *average* dilution below the pulp line. The variation of the settling rate with the initial dilution is in general roughly linear, the rate being proportional to the dilution minus a constant. The type equation is:

$$R = K(F - X) \tag{1}$$

where R = settling rate; K, constant; F, initial dilution; X, constant. The majority of pulps settle according to this formula within the experimental error, at least over a considerable range of dilution. With other pulps, the term $F - X$ must be taken to some power, which may be greater or less than unity. If the data are to be expressed over a wide range of dilutions, more complicated formulas may be required, since the graphs may be more or less S-shaped.

Whether a coincidence or not, it often happens that the constant X in formula 1 is approximately equal to the final dilution. This points to another field for study which would lead to a better understanding of the theoretical principles underlying thickening.

Because X in equation 1 is not always equal to the final dilution and the graphs are not always linear, any accurate selection of a thickener size must be made from zone tests; i.e., the results of settling tests at a number of initial dilutions distributed between the feed dilution and the critical point. The unit area or areas indicated per ton of dry solids per 24 hr. will vary from one initial dilution to the other in these tests if equation 1 does not hold with X equaling the final dilution, since the unit area equals[8]

$$\text{Unit area} = \frac{1.333(F - D)}{R \times dl} \qquad [2]$$

where D = final dilution or pumping dilution and dl = density of the liquid.

When appreciable variation occurs, it is generally the lower dilutions of the free-settling zone that gives the highest unit area. The highest unit area is the one that must be taken, since this is the one that may limit the capacity of a thickener. Coe and Clevenger present data that show such a case. The reported capacity in pounds per square oot per hour was 11.8. The depth of clear solution was 0 ft., indicating that the thickener was working at maximum capacity. The zone test showed capacities all the way from 29 lb. per sq. ft. per hr. down to 8.29 lb. near the critical dilution. It is significant that the maximum actual capacity was much closer to the minimum than to the maximum capacities given by the zone tests. On the other hand, they also present data in their Fig. 9 in which the practical capacity was apparently determined by the behavior at the feed dilution even though tests at lower dilutions indicated a much lower capacity. Further study is needed to reconcile these apparently conflicting results.

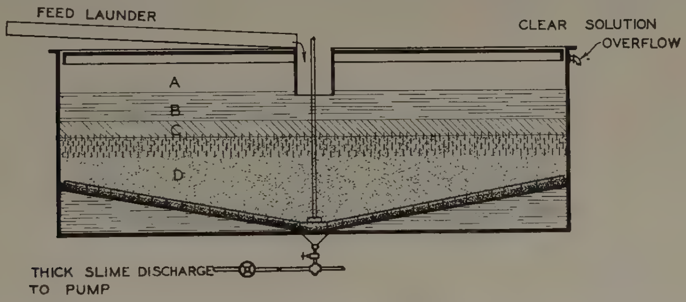

Fig. 3.—Four zones of settling pulp, illustrating continuous thickening (*Coe and Clevenger: Trans. A.I.M.E., vol.* 55).

General Colloidal Aspects

The characteristic of flocs which most commands attention is their elasticity or "all-pervasiveness." A pulp at 10 to 1 dilution properly flocculated apparently fills the liquid completely. At any rate, only a curdlike structure of fine lines is visible. Yet the pulp will settle and thicken to perhaps 1 or 1.5 to 1 dilution, which thick mass may be repulped easily to any desired dilution. Repulped to 6 or 8 to 1, it may look exactly like the 10 to 1 pulp. Of course there is a limit to this apparent expansibility. At very high dilutions clear liquor may be seen between the flocs, as is common in water treatment.

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[8] W. A. Deane: *Trans.* Amer. Electrochem. Soc. (1920) **37**, 659.

Another property of flocs is that of imparting to the pulp a structural viscosity, false body or yield point. That is, the pulp ceases to flow at some finite shearing stress; the pulp is sticky. Now, either due to this general property of the pulp or to the same property of each individual floc, granular particles are entrapped or held up—they do not segregate. A very thick pulp may keep 10-mesh particles in suspension indefinitely. The floc structure is stronger than the force on the particles due to gravity. Although plus 150-mesh or 200-mesh material commonly segregates in an ordinary thickener feed, yet particles as large as 325 mesh are often held by the flocs. This probably aids settlement by weighting the flocs.

Conclusion

Thus, in the thickening of a pulp, colloidal behavior is encountered from start to finish, even though the pulp may contain very little truly colloidal material. In the first place, flocculation of the pulp at least simulates the flocculation of true colloids, if the behavior is not really due to the presence of small amounts of true colloids.

Next, settling in the free-settling zone contains the colloidal elements of floc elasticity and interlocking or structural behavior. The pulp tends to settle as a single unit with collapse at the bottom. This tendency may be adhered to strictly as in type 1 of settling or it may be modified to a partial and progressive collapse all along the pulp column as in type 2. The elastic properties of the flocs enable them to entrap and hold fairly coarse material; also the structural behavior of the pulp as a whole may prevent segregation.

The final phase of settlement or the true thickening step involves the same colloidal elements as the free-settling phase with different emphasis. The elastic properties of the flocs are put to the acid test of squeezing and rearrangement in the compression zone. The structural viscosity or false body of the pulp becomes more defined in this zone and may attain such a degree in some cases as to make the result unpumpable.

Principles of Flotation, I—An Experimental Study of the Effect of Xanthates on Contact Angles at Mineral Surfaces

By Ian William Wark* and Alwyn Birchmore Cox*

(New York Meeting, February, 1932)

In the paper on the development of the flotation process at Broken Hill (Australia) prepared by the Broken Hill Branch of the Australasian Institute of Mining and Metallurgy and published in its *Proceedings* for 1930 (N. S. 80), it is stated that the concentration of ores by flotation has been established on a commercial basis in Australia since 1904 and that differential flotation was practised at Broken Hill in 1912. Since then, progress in flotation practice throughout the world has been rapid, and during recent years the knowledge of the reactions between comparatively modern so-called chemical promoters or collecting agents and the mineral surfaces has been broadened by the published accounts of many investigators, particularly those working in American universities.

Because of the benefits the Australian mining industry has derived from the flotation process, it was felt that the industry should take some part in the efforts that are being made to extend the knowledge of the principles on which the flotation process is based. Accordingly, early in 1930, three Broken Hill mining companies—the Broken Hill South, the North Broken Hill, and the Zinc Corporation—together with the Electrolytic Zinc Co. of Australasia, Ltd., and the Mount Lyell Mining & Railway Co., established a laboratory in the chemistry department of the University of Melbourne for the purpose of investigating the principles underlying flotation. Subsequently, the Burma Corporation, Ltd., expressed a desire to cooperate.

The two authors are directly employed and controlled by these six organizations, and the cost of the investigation is divided equally among them. The research has been carried out under the general direction and in close cooperation with Harry Hey, chief metallurgist for the Electrolytic Zinc Co. of Australasia, Ltd., and the authors desire to place on record their indebtedness to him for his ever willing help and constructive criticism.

After an extensive search of the literature, it was decided to determine in a preliminary investigation whether the effect of chemical collectors could be evaluated quantitatively by the "contact-angle" method. This preliminary investigation showed that with the bubble machine of

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Taggart, Taylor and Ince(1)* quantitative data could be obtained. A description of the precise method used is given later. The investigation is being continued.

SIGNIFICANCE OF CONTACT ANGLE IN FLOTATION

It is obvious that the primary requisite for flotation is sticking between air and mineral, or more strictly, a sufficient attraction between mineral and air for air to be able—partly at least—to replace water at the mineral surface. If replacement of water by air is impossible, flotation as now generally practised is impossible. Other factors influence flotation—mostly, it would seem, by their effect on the froth—but they may be termed secondary in the sense that were contact between air and mineral not possible their influences could not become apparent.

The methods of investigation available for a study of the attraction between air and mineral are many and varied, but we had no hesitation in accepting the measurement of contact angle for the purpose. In the hands of Taggart, Taylor, and Ince(1) the method had recently shown considerable promise and the earlier researches of Langmuir,(2) Edser,(3) and Sulman(4) were also encouraging. Traube and Mishizewa(5) have pointed out the closeness of the connections between angle of contact and wetting, adhesion, adsorption and flotation. If the angle of contact, measured across the water, is zero, there is no wetting (adhesion) by air; if it be 180° there is complete wetting by air. The term "wetting by air" may sound confusing, but it is simply an extension of the term commonly used for oil and other agents. It has the merit of focusing attention on the essential similarity between contacts with air and water respectively. There appears to be no other word that can satisfactorily replace it.

Despite the close connection between angle of contact and floatability, there has been a tendency among modern writers to decry its significance. This is due, perhaps, to the fact that though Sulman and Edser both stressed the importance of contact angle many years ago, no very definite gains have come from its measurement until the recent work of Taggart and his collaborators.

Certain other phenomena have been recognized as being so closely related to flotation that their measurement would give some indication of floatability. Among these are

1. Wettability;
2. Surface tension of the flotation liquor;
3. Capillary rise or fall of the liquor against the mineral;
4. Flocculation of mineral particles.

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* Superior figures in parentheses refer to the bibliography at the end of the paper.

While all of these are closely related to angle of contact, the latter appears to us to be easier to measure and more important. It can be shown that wettability can be evaluated numerically from the contact angle and the surface tension of the liquor. The capillary rise is so closely related to contact angle that it might be used in conjunction with surface tension to evaluate the contact angle. Finally, Bartsch,(6) Sulman,(4) Edser(3) and others have demonstrated that the type of flocculation of value in flotation is that in which several mineral particles are grouped about a central air bubble. The primary condition for its occurrence, therefore, is sticking between mineral and air, and an examination of this sticking through the medium of contact angle is closer to the fundamental than would be a study of flocculation. Edser and Bartsch agree that the cause of sticking is not necessarily electrical. Nevertheless, a study of the connection between contact angle and surface potentials is desirable.

The apparatus developed by Taggart, Taylor and Ince(1) is suitable for the measurement of contact angles. Their interpretation, however, was not truly quantitative. The authors attempted to derive a "collecting index," but the term, which is defined in an arbitrary manner, has neither theoretical nor direct practical significance. However, the contact angle can be used in deriving an estimate of the tenacity of sticking between mineral and air, so that if it can be measured satisfactorily, the primary tendency to float can be evaluated. It is, in fact, for any single mineral, proportional to T (1-cos θ) per unit area of contact, T being the surface tension of the liquor, and θ the angle of contact measured across the liquid phase. For selective flotation, however, a reagent may be of more value than this expression indicates. Thus, a reagent which gave an angle of contact of 90° both with galena and with sphalerite, would, in the absence of modifiers, be of less value than one which gave, say, 50° with galena and zero with sphalerite.

One of the major difficulties in estimating contact angle is that it varies according to the condition of the surface. At a surface over which water is advancing, the angle is greater than that at a surface from which water is receding. The difference between the maximum and minimum values has been termed the "hysteresis" by Sulman. It can be shown that hysteresis is due to friction and that it can be eliminated by suitable means. An equilibrium contact may then be obtained.

The angles cited by Taggart, Taylor and Ince are considerably lower than those we have obtained; part of the difference may be due to hysteresis. These authors introduced another source of uncertainty, as explained later, in their use of cleaved surfaces of galena. Though it is necessary for us to point out the uncertainties in their measurements and to question the adequacy of their interpretation, we, perhaps better than others, appreciate the value of their contribution to the methods of flotation experimentation in introducing what they call the bubble machine.

Other investigators who may be cited under this heading are Edser,⁽⁷⁾ Moses,⁽⁸⁾ Bartell and Osterhoff,⁽⁹⁾ Taylor and Bull,⁽¹⁰⁾ Rideal,⁽¹¹⁾ Bashforth and Adams,⁽¹²⁾ Worthington,⁽¹³⁾ and Gaudin and Martin.⁽¹⁴⁾

Apparatus for Measuring Contact Angles

An air-mineral-water contact is effected at a horizontal mineral surface by bringing a small bubble of air downward into contact with the surface of a small submerged specimen of the mineral. The bubble is suspended at the bottom end of a vertical glass tube of small diameter. A magnified image of the contact is projected on to a photographic plate on which the angle of contact is measured easily.

The design of Taggart, Taylor and Ince's bubble machine⁽¹⁾ has been closely followed. The diameter of the bubble holder is 3 mm. and of the bubble about 5 mm. The glass cell containing the mineral is mounted on a mechanical stage which admits of movement both in the direction of the optical axis of the system and across it. The bubble holder is attached to a second mechanical stage, which enables it to be raised or lowered or to be moved for focusing along this axis. The bubble may thereby be conveniently brought into contact with any point on the surface of the mineral.

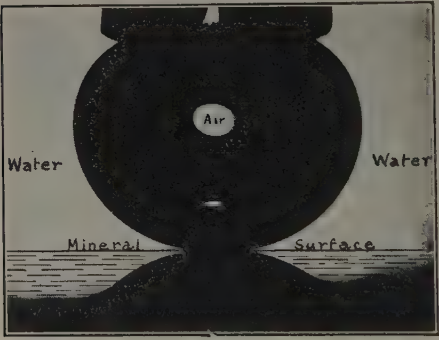

Fig. 1.—No contact between air and mineral. × 12.
Note reflection of air bubble in mineral surface.

It has been found desirable to place the lamp in a plane slightly higher than that of the surface of the mineral. Light is then reflected from the mineral surface and the image of the bubble on the screen is seen in juxtaposition with an inverted image which arises by reflection at the surface of the mineral. The accompanying photographs illustrate how this facilitates the measurement of the angle.

Fig. 1 shows a bubble pressed down on a surface with which it does not coalesce. It might appear that there was a contact angle of something under 10°, but on raising the bubble there was no tendency for the bubble to cling, as invariably there is if contact has been effected. The photograph illustrates the difficulty of measuring angles below 10° with any degree of certainty.

It is unnecessary to make a photograph in every case. The angle may be measured with sufficient accuracy on the ground-glass screen of the camera, using a transparent protractor.

Care must be taken to see that the angle measured is the true equilibrium value. For this reason vigorous tapping of the stage upon which the cell rests is necessary. Tapping has been found necessary by earlier workers,(2,15,19) both for the measurement of contact angle and of surface tension.

The angles corresponding to the two sides of a bubble are approximately equal, but there is usually a difference of one or two degrees, and this may on rare occasions amount to ten degrees or more. Though it is probably unnecessary, it has been customary on such occasions to make the bubbles more uniform by movement of the stage and again tapping. Ablett's work(15) proves that the mean of these two angles is nearly identical with the equilibrium angle. (The refinement, suggested later, of using the cosine mean is not justified by the degree of accuracy of our measurements.) It has been customary, therefore, to measure the angle on both sides of the bubble. At least three positions on the mineral surface are examined; any recorded angle is therefore the mean of at least six measurements.

Small variations in the size of the bubble have little or no influence on the angle of contact.

The same angle of contact is obtained by use of a drop of the liquid in air instead of a bubble of air in the liquid. It is, of course, necessary to measure the angle across the liquid in each case.

The procedure adopted has been kept uniform. After contact has been effected the bubble is pressed down on the surface, the holder is then raised until the area of contact just begins to contract, and the table is tapped until a constant angle of contact is obtained.[1] Tests have shown that this value is not much altered by the actual procedure. The same value is obtained if the table is tapped while the bubble is pressed down on the surface, but a longer time of tapping is necessary before the equilibrium angle is reached. The readiness with which equilibrium is established is noticeably a function of the smoothness of the surface.

PREPARATION AND PURIFICATION OF XANTHATES

The need for purification of xanthates for use in work of the type now under discussion is obvious. Fortunately, the dixanthogens and carbon bisulfide are ether-soluble, whereas the xanthates are not. Recrystallization (generally twice) from alcohol followed by an ether extraction of the solid has therefore been adopted. Xanthates frequently contain 10 per cent of ether-soluble compounds such as dixanthogens and 10 per cent of solids insoluble in alcohol. The latter are not flotation agents, but the former probably interfere with the selectivity of the xanthate. For the preparation of those of the above xanthates unobtainable in quantity the methods described by Foster(16) were adopted with only minor modifications.

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[1] Edser(3) has stated that, for spherical bubbles, "When the angle of contact is too large it will decrease quickly until stable equilibrium is attained, and when it is too small it will increase fairly slowly." This explains our adoption of this procedure.

The potassium methyl xanthate was prepared by slowly adding a slight excess of carbon disulfide to a saturated solution of potassium hydroxide in methyl alcohol, keeping the temperature below 20° C. A pasty mass of yellow crystals rapidly formed.

The benzyl, phenyl ethyl, cyclohexanyl, and cetyl xanthates were prepared by slowly adding excess carbon disulfide to a mixture of the respective alcohol with a slight excess over the theoretical amount of a saturated solution of potassium hydroxide in water. The mixture was kept at approximately 20° C. until all the carbon disulfide was added; stirring was continued for 15 min. and the pasty mass was then warmed in a water bath at 50° C. Finally, excess carbon disulfide was removed under reduced pressure by means of a water pump. The yellow crystalline mass was then dissolved in as small a quantity of warm acetone as possible, decanting from the small residual aqueous solution of red polysulfides. The addition of petroleum ether precipitated the xanthates as pale yellow crystals.

The cetyl xanthate required a slight modification of the foregoing procedure. As cetyl alcohol is a solid, it was necessary to use it as a solution in petroleum ether. The reaction was slower and the mixture was warmed for 15 min. in a water bath at 50° to complete the reaction. The product crystallized without addition of petroleum ether from warm acetone as very pale yellow flakes, almost white.

Preparation of Clean Mineral Surfaces

The surfaces of the ground minerals that enter the flotation machine are produced either by cleavage or by grinding. It may be assumed that the greater part of the total surface has been produced in the presence of the liquor of the grinding circuit. A small percentage, nevertheless, must consist of surfaces which have been weathered by long exposure in the cracks of the orebody or by shorter exposure since breaking. To approximate flotation practice as closely as possible, two methods of surface preparation needed investigation; namely, cleavage of the pure mineral under water and grinding of the pure mineral under water. Fortunately, the angles of contact at clean surfaces of both types have proved identical. Neither method is free from experimental difficulties.

Cleavage.—Cleavage tends to follow old cracks; it is therefore only one of many attempts that produces a clean mineral surface. Often a surface that appears clean must be rejected, frequently a surface tarnish indicates incipient oxidation or other weathering. Only when the cleavage is effected under water is the surface free from contamination. There is, moreover, difficulty in producing flat surfaces by cleavage. Galena, certain rare specimens of sphalerite, and one or two gangue minerals are the only minerals that give sufficiently flat surfaces for this method of testing.

Grinding.—A ground-glass plate is satisfactory for grinding, and by using a coarse plate no further treatment of galena may be necessary. Generally, however, this treatment leaves the surface coated with very fine slime particles of the mineral, which render it unsuitable for general use. The slime may be removed from a galena surface by wiping the submerged surface with linen or filter paper. The surface then behaves in a manner identical with a clean cleaved surface, and actually consists of a series of cleavage faces.

Polishing.—Though grinding proved satisfactory for galena it was suitable for sphalerite only on rare occasions. With this mineral the surface could not be freed from slime in any simple manner. Since polished surfaces are generally free from contamination by slimed particles, such surfaces were tested.

The method of polishing was that usually adopted for the preparation of mineral surfaces for microscopic examination, except that the surface was finally wiped on clean linen under water to remove adhering particles. An undressed linen, thoroughly digested with water, was used. No amount of rubbing on linen alone causes the development of an angle of contact in distilled water. This method of polishing has proved entirely satisfactory, not only for sphalerite, but also for all the minerals with which we have experimented. The tests described later have been carried out with polished surfaces unless there appears a statement to the contrary.

Table 1 demonstrates that, provided the surface is freshly prepared and is free from slime, the actual method of preparation is immaterial.

TABLE 1.—*Galena: Angle of Contact in 25 Mg. per Liter Potassium Ethyl Xanthate Solution*

Preparation of Surface	Angles of Contact in Independent Experiments, Deg.
Ground on coarse glass under running water	56, 58, 56
Ground on coarse glass under distilled water	60, 58, 59.5, 61.5
Ground on grooved steel plate under distilled water	59 (same after wiping on linen 57)
Ground on glass plate and wiped on filter paper	57–60, 56
Ground on glass plate and wiped on linen	58, 58, 59.5, 60, 59.5, 59, 59
Scraped with steel blade after grinding level on glass	60, 60
Surface formed by cleavage under water	66, 56, 64, 62
Polished and wiped on linen	60, 60, 57, 60, 59, 59

The over-all average is 59°, the maximum deviation 3°, except for cleaved surfaces, at which measurement is most difficult.

Test of Cleanliness.—Several hundred tests prove that there is no tendency for a bubble of air to replace water at the surface of any of the common zinc, lead, iron or copper minerals or at surfaces of the associated gangue minerals. Each surface, therefore, is tested in this way before it is used in determining the effect of any reagent or condition.

Langmuir(2) is in agreement with our contention that clean sulfide surfaces are readily water-wetted. Weinig and Palmer(17) and Sulman(4) state that fresh cleavage faces of calcite, sphalerite, galena, pyrite and magnetite are wetted by water. Ince(18) says "The bubble showed the very slight adhesion and distortion common to slightly contaminated sulfide surfaces." This implies that uncontaminated surfaces would exhibit a zero angle. Taggart, Taylor and Ince(1) say, "Since it is difficult to get mineral particles so clean that they show no tendency at all to attach to air bubbles . . . " This is further evidence on the same point. One finds it difficult, therefore, to understand why Taggart and his collaborators commenced tests with galena crystals already showing contact angles up to 34° in distilled water, especially as, with other crystals, the angle, after addition of what they deduced were good collectors, amounted sometimes only to 27°. This introduces an unfortunate source of uncertainty in all their results, which is best demonstrated by an example:

Quoting from their paper, with one galena specimen dichlorbenzene made no difference to the angle of contact in distilled water (34°). With another, showing an angle of 19° in distilled water, addition of amyl mercaptan caused an increase to 33°. Had the latter reagent been tested with the first specimen, it appears doubtful whether it would have caused any increase. Our own work has shown that the increase in angle caused by a reagent is not independent of the initial condition of the surface. Figures for sphalerite surfaces conditioned by copper sulfate and showing a zero angle in distilled water are:

	Degrees
A. Angle in 25 mg. per liter potassium ethyl xanthate	60
B. Angle in 25 mg. per liter potassium amyl xanthate	84
C. Angle when the specimen from *A* was placed in solution *B*	73
rising slowly probably to	84

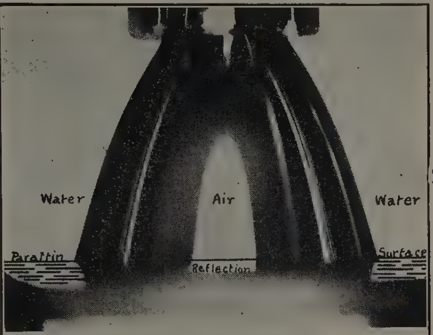
The gradual rise in *C* corresponds to a slow substitution of amyl xanthate for ethyl xanthate at the surface of the sphalerite. The difference method of Taggart and his associates, if applied in this case, would give the low rating of 13° to amyl xanthate. It is not contended that the authors would have been content to use it in such a case; evidently they rejected surfaces showing an angle of contact much above 30° in distilled water. It is clear, however, that the only satisfactory method is to commence with uncontaminated surfaces.

RELIABILITY OF METHOD

Probably the most satisfactory manner of testing the method is to compare results from it with those of other workers who have been able to use more accurate, though less expedient methods. Reliable results are given for the angle of contact at a paraffin surface by Bosanquet and Hartley⁽¹⁹⁾ (106° 28′), Ablett⁽¹⁵⁾ (104° 34′); Coghill and Anderson⁽²⁰⁾ (105°).

The greatest weight must be given to Ablett's result, which was obtained at 10° with Kahlbaum's wax melting at 56.7° C.

Using Merck's wax of 56° to 58° melting point, our own determinations have averaged 106° at 16° C. The agreement is good enough to demonstrate that angles of contact can be estimated with sufficient accuracy by the method adopted. A photograph of the contact angle is included (Fig. 2). This degree of agreement proves further that no buoyancy correction is necessary in our estimate of angle of contact, a conclusion which is further supported by the close agreement between angles of contact measured with air bubbles freely suspended and held by the glass holder respectively.



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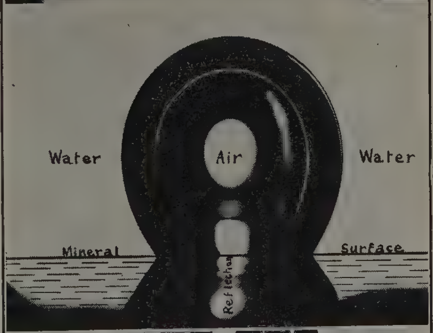

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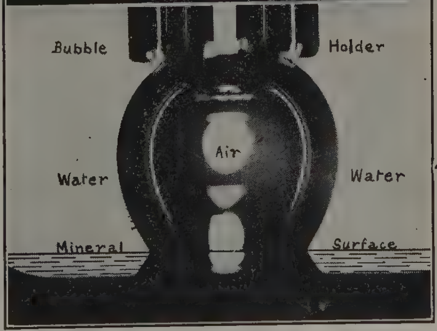

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FIG. 2.—CONTACT BETWEEN AIR AND PARAFFIN. × 12.

FIG. 3.—CONTACT BETWEEN ETHYL XANTHATE COATED SURFACE AND AIR. FREELY SUSPENDED AIR BUBBLE. × 12.

FIG. 4.—CONTACT BETWEEN ETHYL XANTHATE COATED SURFACE AND AIR. BUBBLE HELD DOWN BY HOLDER. × 12.

With a paraffin surface, the angle for a freely suspended bubble was 104° against 106° using the holder. The difference is not significant. Many other measurements at a galena surface in ethyl xanthate solution, where the angle is about 60°, indicate no significant difference in contact angle corresponding to the two methods of attachment. Fig. 3 is a photograph of a bubble freely attached to a galena surface in the presence of 25 mg. per liter ethyl xanthate. Fig. 4 is a photograph of the normal method of attachment to a galena surface in the same solution.

Reproducibility.—The following sets of figures, taken at random from a notebook, show how uniform the angle is over the surface of the mineral specimen:

I. Polished chalcocite in potassium ethyl xanthate solution, 25 mg. per liter, Sept. 16, 1930.

Angle on left side	60	60	57	60	
Angle on right side	62	60	59	62	
Average	61	60	58	61	Over-all average, 60°

II. Polished cleiophane activated by copper sulfate March 7, 1931, in potassium amyl xanthate solution, 25 mg. per liter.

Angle on left side	90	85	84	
Angle on right side	84	88	85	
Average	87	86.5	84.5	Over-all average 86°.

Two observers generally agree within one degree in the measurement of an angle of contact.

Two similar specimens in the same or similar solutions give angles of contact within one or two degrees. Several scores of measurements for galena in solutions of potassium ethyl xanthate, 25 mg. per liter, have, in the most recent work, shown angles of contact between 58° and 62°.

Effect of Ethyl Xanthate on Various Minerals

Except where a statement to the contrary appears, the potassium salt (referred to in the sequel as KEtX) at a concentration of 25 mg. per liter has been used; the pH value of the solution has been between 6 and 6.5 and tests have been carried out at room temperature.

Sulfides

Galena.—Table 1 shows that the characteristic angle of contact for galena from the Zinc Corporation mine, Broken Hill, is 59°. The same angle is characteristic of galena from several other sources. Specimens have been tested from Broken Hill South and North Broken Hill mines, New South Wales; Rosebery and Zeehan, Tasmania; Northampton, Western Australia, and from North America. All of the samples appeared pure to visual examination but the microscope generally revealed the presence of small inclusions of impurities of which tetrahedrite was the most common. One specimen from Rosebery assayed 99 per cent PbS; this was probably the least pure of the specimens.

There was a definite variation in the physical structure of the specimens. Those from Tasmania were of fine grain; those from Broken Hill readily cleaved into large cubes.

To determine whether there was any difference between surfaces parallel with and diagonally across cleavage planes, a galena crystal was ground so that the surface was equally inclined to all three cleavage planes. It then appeared much finer grained than usual; the angle in a xanthate solution was 58°, against a mean value of 59° for some scores of measurements on surfaces parallel to cleavage planes.

Some crystals of Broken Hill galena were fused in vacuo in a silica tube; upon solidification a very fine-grained specimen was obtained at the surface of which the angle of contact was 57.5°. As might have been anticipated, the angle of contact is unaffected by the particular ethyl xanthate used; the sodium, potassium, and lead salts gave identical angles, though the lead salt was not soluble enough to give a 25 mg. per liter solution.

Sphalerite.—Neither in distilled water[2] nor in an ethyl xanthate solution is there any tendency for an air bubble to stick to a sphalerite surface. An activator must be added before sticking will occur. This result is in agreement with the direct flotation experiments of Gaudin, Haynes and Haas.(21) The reactions of activators are discussed later; in this section it will be assumed that the sphalerite has been activated by contact with a solution containing copper sulfate or other heavy metal salt before the addition of the xanthate.

The measurements of the angle of contact at activated sphalerite surfaces are summarized in Table 2.

TABLE 2.—*Angle of Contact at Sphalerite Surfaces in 25 Mg. per Liter KEtX Solution*

Source of Sphalerite	Surface Preparation	Activator	Time of Contact with Xanthate Solution	Contact Angle, Deg.
Joplin, Mo.	Cleaved under water	20 mg. per liter $CuSO_4.5H_2O$		60 (Mean of 5 tests)
Joplin.	Polished	25 mg. per liter Ag_2SO_4	10 min.	58
Swansea, Tasmania.	Polished	25 mg. per liter Ag_2SO_4	10 min.	57
	Polished	15 mg. per liter $HgCl_2$	10 min.	59
	Polished	10 mg. per liter $CuSO_4.5H_2O$	24 hr.	59
	Polished	30 mg. per liter $CuSO_4.5H_2O$	24 hr.	60
	Polished	10 mg. per liter $CuSO_4.5H_2O$	30 min.	61
	Polished	7 mg. per liter $CuSO_4.5H_2O$	30 min.	59
	Polished	2 mg. per liter $CuSO_4.5H_2O$	10 min.	61
New Jersey (Cleiophane). .	Polished	10 mg. per liter $CuSO_4.5H_2O$	30 min.	61

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[2] To insure absence of heavy metal salts from the distilled water employed in all the tests described in this paper, redistillation in glass apparatus was adopted. Despite the low solubility of the heavy metal xanthates, no precipitate was ever obtained on making up the xanthate solution. The dimethyl glyoxime test showed that the copper content of the redistilled water was less than 0.01 mg. per liter.

Work with amyl xanthate has revealed no difference in the contact angle at surfaces of cleiophane (iron-free blende), resin blendes from Rosebery and Joplin, and marmatite from Broken Hill. It is not implied that modifying agents would be without differential action, especially if they reacted differently with sphalerite and pyrrhotite.

Iron Minerals.—(*a*) *Pyrite.*—Specimens from Mt. Lyell and Rosebery, Tasmania; from Leadville, Colo., and from an unknown source all gave identical angles in xanthate solution. The maximum angle attained with pyrite in 25 mg. per liter KEtX solution has been 61°; owing to the rapid dissolution of iron and consequent precipitation of iron xanthate on the surface, however, effective contact is not possible for long after the initial contact. If the surface is cleaned by wiping with linen, full contact becomes possible again. Angles determined in successive tests on various specimens were 59°, 59°, 59°, 61°, 58°, 61°, 59°, 58°, and 60°, an average of 59.5°.

(*b*) *Pyrrhotite.*—Pyrrhotite from only one source (Broken Hill) was tested. Neither in distilled water nor in an ethyl xanthate solution was there any tendency for an air bubble to stick to the surface. When the surface was activated by 10 mg. per liter $CuSO_4.5H_2O$, however, ethyl xanthate led to a degree of sticking corresponding to angles of 58°, 60° and 59° in three successive experiments.

Fouling of the surface by precipitated iron and copper xanthates is troublesome. After long standing, not only is the surface coated in this manner, but it is also tarnished to a dull bronze color. Mere wiping with linen is insufficient to restore contact with air, nor can this be obtained by further additions of copper sulfate and/or xanthate. Pyrrhotite tends to lose its response to xanthate more readily than any of the other sulfide minerals.

Copper Minerals.—Specimens of bornite were obtained from Mt. Lyell and from the Transvaal. Those of chalcopyrite were obtained from Mt. Lyell, Broken Hill and Canada. The covellite specimen was of unknown origin and the three chalcocite specimens were from Cobar, N. S. W., from Montana, and from an unknown source. The latter was associated with malachite. The tetrahedrite was from Rosebery.

Early work was somewhat baffling because contact angles were not as steady with regard to variations in time of contact as had been those with other minerals. Gradually, however, a regularity was noticed in the variations. When equilibrium was reached the angle appeared to have one of two possible values, which were 60° and 80° respectively. Intermediate angles were obtained, but these were not stable. Irregularities over the surface were not uncommon, angles of 60° and 80° being obtained at different parts of the same surface. The higher angle was obtained more often in concentrated solutions and once, when a chalcopyrite specimen had exhibited it, a galena specimen in the same solution also gave it.

The reasons will be discussed later for the ultimate conclusion that the high angle is due to a decomposition product of the xanthate; namely, dixanthogen. It will suffice to state here that several scores of measurements have shown that when the conditions are suitable the dixanthogen effect is not observed, and that then the stable angle of contact lies between 58° and 62° for the five minerals.

Some of these minerals tend to pass readily into solution and/or to tarnish rapidly after polishing. These changes are to be attributed in general to oxidation of the surface layer, and they result in a variable concentration of copper ions in solution. Variations in the rate of solution, which occur with apparently identical specimens of the same mineral, have not been accounted for, but it is considered that they may affect the contact angle in three ways: (1) It is generally accepted that cupric xanthate—which would be formed by interaction between dissolved cupric ions and xanthate ions—is unstable, the decomposition products being the cuprous salt and dixanthogen; (2) unless there is an appreciable copper-ion concentration, it has been found that xanthate will not condition a chalcopyrite surface. If enough copper has not dissolved by natural processes, some must be added to induce sticking. Such addition has not been found necessary for ethyl xanthate, but is necessary for methyl xanthate and some other reagents. A very small quantity suffices. This observation, which is of considerable importance to the theory of the action of xanthates, will be discussed elsewhere. (3) The precipitated xanthate (presumably cuprous xanthate) tends to foul the surface, preventing proper contact between mineral and air. Since the precipitate itself is readily floatable, it may be removed from the surface by means of the bubble, and contact then becomes normal. It may also be removed by wiping the surface with linen. It is difficult to predict the importance of this effect in flotation. The violent agitation in the pulp would tend to rub off any such surface coating that was not directly floated away by the air bubbles.

Gangue Minerals

The gangue minerals tested were calcite from North Broken Hill mine, rhodonite ($MnSiO_3$) from Zinc Corporation mine, quartz from Zinc Corporation and Broken Hill South, and schist from Rosebery and Mt. Lyell.

No contact occurs between an air bubble and polished surfaces of any of these minerals either in distilled water or in xanthate solution. An increase in the xanthate concentration to 500 mg. per liter and/or the addition of 20 mg. per liter $CuSO_4.5H_2O$ does not induce contact. Cleavage surfaces of calcite and rhodonite are equally inert.

The gangue minerals are thus to be placed in a different category from the sulfides which, even if unaffected by xanthate alone, become air-avid in the presence of copper sulfate. Though xanthate is without effect on these gangue minerals, it does not follow that other reagents are without effect. Indeed, it is known that oleic acid has some action on calcite.[22]

Nonsulfides

Anglesite ($PbSO_4$).—Samples of anglesite were obtained from Broken Hill and Rosebery. The solubility of anglesite is much greater than that of any of the sulfide minerals (it is reported to be 0.042 g. per liter at 20°) and, consequently, upon the addition of xanthate a copious precipitate forms. It is not surprising, therefore, that contact between air and mineral cannot be effected by low xanthate concentrations. With 100 to 200 mg. per liter of xanthate contact can be effected.

As xanthate is added to a saturated $PbSO_4$ solution much of the lead ion is removed as the less soluble lead xanthate; the xanthate ion is also removed, presumably before it has access to the solid surface. More $PbSO_4$ dissolves to maintain saturation of the solution and further xanthate addition removes more lead from solution, with consequent increase in sulfate-ion concentration. The main reaction, therefore, is

$$\underset{\text{solid}}{PbSO_4} + 2X^- = \underset{\text{solid}}{PbX_2} + SO_4^{--}$$

When this reaction comes to equilibrium, the lead xanthate and sulfate-ion concentrations must be such that $[Pb^{++}][SO_4^{--}]$ = constant = solubility product of $PbSO_4$ and also $[Pb^{++}][X^-]^2$ = constant = solubility product of PbX_2. This condition is reached only when the Pb^{++} concentration is considerably lower than that corresponding to a pure $PbSO_4$ solution. The $[SO_4^{--}]$ is correspondingly high. This sulfate-ion concentration builds up only to half the extent that xanthate ion has been added.

This appears to be rather an expensive way of bringing the SO_4^{--} concentration to the necessary value. It is probable that initial additions of sodium sulfate would reduce the consumption of xanthate. Qualitative tests with single crystals of anglesite tended to confirm this deduction in that sodium sulfate decreased the amount of lead xanthate formed. It is impossible, however, to deduce from these tests whether additions of sulfate would lessen consumption of xanthate in the flotation of oxidized ores.

In the experiments carried out, angles of contact of 61°, 58°, 58°, 56° and 59° have been obtained.

Cerussite ($PbCO_3$).—A specimen of cerussite from Broken Hill was used. Again the solubility of the mineral is higher than that of galena and higher xanthate concentrations are necessary. Some sticking appears to occur with a low xanthate concentration (25 mg. per liter) but as the lead xanthate formed in the solution settles on the surface, sticking ceases to be possible. This is best revived by addition of more xanthate; wiping alone is insufficient to induce sticking. The contact angle in a 2 g. per liter KEtX solution was 57°.

Crocoite (*$PbCrO_4$*).—A specimen of crocoite from the Zeehan district was used. The surfaces available were smaller than is desirable for exact measurements of contact angle. No contact was possible in 25 mg. per liter KEtX solution, but at 2 g. per liter KEtX the angle of contact was 63°. No intermediate concentrations were tried. On standing, a heavy precipitate of lead xanthate was obtained; wiping removed this and contact was again possible.

Azurite ($2CuCO_3.Cu(OH)_2$).—Only a small specimen of azurite was available and this contained inclusions of malachite and unidentified gangue mineral. There is no tendency for contact to occur between air and mineral in distilled water. In a 200 mg. per liter ethyl xanthate solution the contact angle was between 50° and 66° after 30 min. contact (accurate measurements were impossible with this specimen) and after two days it was much higher (70° to 75°). The high angle is probably to be attributed to the formation of dixanthogen, as is the case with the copper sulfide minerals. A better specimen is necessary for the accurate determination of contact angle. This test is valuable, however, in proving that contact is possible under some conditions.

Effect of Other Xanthates on Various Minerals

In the aliphatic series, xanthates corresponding to the following alcohols have been investigated:

Formula	Alcohol	
CH_3OH	Methyl	
C_2H_5OH	Ethyl	
C_3H_7OH	Propyl	
C_4H_9OH	Butyl	(normal and iso-compound)
$C_5H_{11}OH$	Amyl	(iso-compound)
$C_{16}H_{33}OH$	Cetyl	

and in the cyclic series xanthates corresponding to

Formula	Alcohol
$C_6H_5.CH_2OH$	Benzyl
$C_6H_5.C_2H_4OH$	Phenyl ethyl
$C_6H_{11}.OH$	Cyclohexanol
$C_{10}H_{17}.OH$	Fenchyl

Originally, it was intended to prepare other xanthates. It now seems probable that no greater selectivity will be obtained with other members of the series, unless through the simultaneous use of modifiers. This conclusion is drawn because the solubilities of the heavy metal xanthates have remained in the same order whatever xanthate has been taken. Greater selectivity can only be expected if the introduction of the organic radicle changes the order of solubility of the metallic salts. A marked change in type of the alkyl or aryl radicle might conceivably change this order.

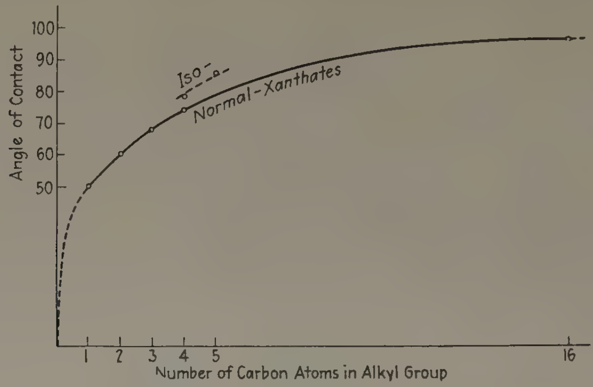

FIG. 5.—DEPENDENCE OF CONTACT ANGLE ON ALKYL GROUP.

The measurements of contact angle for all these xanthates, and for several minerals, are summarized in Table 3. Solutions contained 25

TABLE 3.—*Contact Angles for Various Minerals and Xanthates*[a]

	Xanthate										
	Methyl	Ethyl	*n*-Propyl	*n*-Butyl	iso-Butyl	iso-Amyl	Cetyl	Benzyl	Phenyl Ethyl	Cyclo-hexanol	Fenchyl
Galena.......	50°*X*	59	68	74	77	88	100	71	70	73	72
Sphalerite....	51*X*	61*X*	69*X*	74*X*	79*X*	84*X*	97	71*X*	71*X*	75*X*	73*X*
Pyrrhotite...		59*X*				86*X*	95	72*X*			
Pyrite.......	49*X*	60	67	74	78	82	95	71	70	75	74
Bornite......	51	60	68	73	78	86	95	70	71	75	73
Chalcopyrite	52*X*	60	69	76	78	90	94	75	72	74	73
Chalcocite...		60									
Anglesite.....		58									
Cerussite		57									
Cleiophane...	50*X*	61									
Crocoite.....		63									
Tetrahedrite.		60									
Mean value, degrees	50	60	68	74	78	86	96	72	71	75	73

[a] With the copper minerals the figures given represent the contact angle obtained before dixanthogen begins to form. This generally implies that the measurements be made before 2 hr. contact between mineral and solution, but this time is variable.

mg. per liter of the xanthate, except in the case of the cetyl compound which was used as a saturated solution. Fuller details follow. The sign *X* indicates that activation (with $CuSO_4$ or $HgCl_2$, etc.) was necessary.

The mean results for the aliphatic series are graphed in Fig. 5. Starting, presumably, with a zero angle of contact for sodium dithiocarbonate the angle of contact rises rapidly at first and then more slowly as the length of the carbon chain in the nonpolar group increases. The angle seems to approach asymptotically the limit 104°, which is the value given by a solid paraffin surface. The angles for the more compact iso-compounds are somewhat higher than those for the normal. It is significant that Gaudin and Martin(14) have found the iso-compounds to be the better agents in flotation.

The angles obtained with the four cyclic xanthates lie close together, near to butyl xanthate. This is the position which might have been expected from their other properties.

TABLE 4.—*Contact Angles in* 25 *Mg. per Liter KMeX Solution*

Mineral	Activator	Contact Angle	Time of Reading	Remarks
Galena	None	Nil	Up to 16 hr.	No angle developed even in 125 mg. per l. xanthate or on addition of 10 mg. per 1. $Pb(NO_3)_2$ and/or on wiping the surface
	Various activators, salts of Ag, Hg, Cu	Irregular, maximum 48° to 52° in different tests		Contact is generally difficult to effect, and wiping is always necessary. (See discussion.)
Sphalerite	None	Nil	0 to 90 min.	
	2.5 mg. per l. of Cu^{++}	51°	10 to 60 min.	25 min. contact with $CuSO_4$ before KMeX addition
Cleiophane	2.5 mg. per l. of Cu^{++}	50° 51°	10 min. 16 hr.	Wiping was without effect on the surface.
Pyrite	None	Nil	10 min.	
	2.5 mg. per l. of Cu^{++}	48°	Up to 1 hr.	KMeX added first; then $CuSO_4$.
	2.5 mg. per l. of Cu^{++}	50°	60 min.	$CuSO_4$ first. Wiping by linen was necessary.
Bornite	None	50° to 51°	5 min. to 24 hr.	Contact was immediate. Wiping was without effect.
Chalcopyrite	None	Nil	To 15 min.	
	2.5 mg. per l. of Cu^{++}	50° to 52°	Up to 90 min.	Wiping necessary to obtain uniform sticking of bubble all over surface
		59° 73°	24 hr. 72 hr.	Explanation in discussion

Discussion of Effect of Various Xanthates on Minerals

In the following detailed description only abnormal results and difficulties are stressed. Only on rare occasions are such difficulties encountered.

Methyl Xanthate.—The results for various minerals are given in Table 4, which illustrates the types of difficulties encountered with a troublesome reagent.

Difficulty was experienced in obtaining the true angle of contact with galena. Though the mineral can be activated by Cu^{++}, Hg^{++} and Ag^{+}, the reaction does not appear to progress rapidly, and often activation is irregular. On some occasions, however, activation was satisfactory, angles of 52°, 48° and 50° were obtained under the following conditions:

1. With 5 mg. Cu per liter and 50 mg. per liter KMeX after contact for 1 hr. and wiping on linen.
2. With 7½ mg. Cu per liter and 25 mg. per liter KMeX after 75 min. contact and wiping on linen.
3. With 15 mg. Hg per liter and 25 mg. per liter KMeX immediately after the xanthate addition. Half an hour later, owing to the settling of the precipitated mercury xanthate, contact was not possible, but wiping restored contact (49°).

The last test suggests that the cause of nonsticking and/or irregular sticking is settling of the precipitated mercuric, cupric or silver xanthates on the surface.

It will be noticed that pyrite, galena and sphalerite require activation by a heavy metal salt. Bornite, of all the minerals tried, alone responds

Table 5.—*Contact Angles in 25 Mg. per Liter K Propyl X Solution*

Mineral	Activator	Angle of Contact, Deg.	Time of Reading, Min.	Remarks
Galena......	None	64	10	
		67	45	
		68	100	
		70	1 day	
Sphalerite....	10 mg. per l. $CuSO_4.5H_2O$	66	5	
		69	35	
		(86)	1 day	Dixanthogen effect (Note presence of copper as activator)
Pyrite.......	None	67	25	Full angle developed within 1 minute
		67	60	
Bornite......	None	65	10	
		68	50	
Chalcopyrite.	None	69	15	Dixanthogen effect noticed very early
		(78)	40	

to methyl xanthate without activators. Chalcopyrite fails to respond to methyl xanthate, unless there is a certain minimum copper-ion concentration in the solution. Though this mineral gives a normal angle at first, a higher angle develops later, which may be attributed to the formation of methyl dixanthogen.

n-Propyl Xanthate.—Results for various minerals are summarized in Table 5. The results for this reagent are typical of those for the higher xanthates, which give little trouble.

n-Butyl Xanthate.—The characteristic angle is 74°. It is unnecessary to set out results in detail since they closely resemble those of Table 5. Great care was necessary to prevent the development of the dixanthogen angle. The solid xanthate decomposes on standing. A recrystallization and ether washing just prior to the test is necessary. Since the solution decomposes readily, giving an emulsion of the dixanthogen, a fresh solution must be made for each test.

Iso-butyl Xanthate.—The characteristic angle is 78°. Again it is unnecessary to include details. With the copper minerals and with sphalerite activated by copper sulfate there is a tendency to develop the dixanthogen angle. This for the butyl compounds seems to be higher than that for ethyl dixanthogen. Both butyl dixanthogens lead to angles of 84° to 88°.

Iso-amyl Xanthate.—The characteristic angle is 86°. The results cited in Table 3 were obtained at an early stage in this investigation, which probably accounts for the rather large variation from the mean value. The development of the equilibrium angle is slower. Generally, the angle has reached only about 75° after 15 min., and 2 hr. is required for the development of the full angle. With pyrrhotite a rapid dissolution of iron occurred, resulting in a heavy xanthate precipitation. Only when this was removed by wiping the surface with linen was the true angle obtained. Chalcopyrite decomposed the amyl xanthate at a fast rate; the amyl dixanthogen angle seems to be close to 100°.

Cetyl Xanthate.—The characteristic angle is 96°. As the solubility of the cetyl xanthate is less than 25 mg. per liter, a saturated solution was used. No activators were necessary for any of the minerals. The surfaces of pyrrhotite and chalcopyrite became coated with precipitates of iron and copper xanthates respectively; to obtain the true angle wiping with linen was necessary. Galena was exceptional in exhibiting a second (unstable) contact angle of 75°. On several occasions different points on the same surface showed angles of 75° and 95° respectively.

Benzyl Xanthate.—The characteristic angle is 72°. With the exception of chalcopyrite, all the minerals gave angles between 70° and 72°. The higher angle for chalcopyrite, which rose to 84° overnight, presumably is due to the slow formation of benzyl dixanthogen.

Phenyl Ethyl Xanthate.—The characteristic angle is 71°. After overnight contact with 25 mg. per liter phenyl ethyl xanthate, both chalcopyrite and pyrite gave contact angles of 84°, which presumably is due to dixanthogen formation. Angles lay between 70° and 72° for all the minerals after contact for 1 hr. with the solution. The solubility of the zinc salt is such that sphalerite lies in a critical region where an activator must be added with low concentrations of xanthate, but where slight and irregular sticking between mineral and air may occur with higher concentrations and without the addition of any heavy metal salt.

Cyclohexanyl Xanthate.—The characteristic angle is 75°. In addition to the minerals tabulated in Table 3, pyrrhotite was tested. Owing to the rapid deposition of copper and iron xanthates on the surface by settling of precipitates in the solution, no satisfactory measurement was possible. This failure probably was due to the condition of the particular specimen of pyrrhotite used, which dissolved with abnormal rapidity in the solution. The copper xanthate was formed from the sulfate, which had to be added as an activator.

Fenchyl Xanthate.—The original commercial sample was difficult to purify, the first "purified" sample retaining enough impurity to cause the development of an angle of contact of 83° under certain conditions. This was particularly noticeable with sphalerite when activated by copper sulfate, but not by silver sulfate. Further purification yielded a sample which showed no unusual properties, the characteristic angle being 73°.

Effect of Xanthates on Metals

It is of interest in considering the theories of the action of xanthates on the sulfide minerals to note that they are effective collectors not only for sulfide minerals but also for oxidized minerals and for metals themselves. On the other hand, ethyl xanthate is without apparent effect on sulfur. These statements, of course, are based on measurements of contact angle.

Not only are xanthates effective for the metals, but the angle of contact is apparently identical with that obtained for the sulfide minerals if the dixanthogen effect be eliminated. It is difficult to eliminate this effect, for the metals catalyze the production of dixanthogen from the xanthates. For this reason work with the lower xanthates gives abnormally high angles; namely, 74° for both copper and mercury with ethyl xanthates.

It is surprising that activation of zinc metal by copper sulfate, followed by ethyl xanthate treatment, has not led to any development of contact angle.

Figures for higher xanthates are closer to their values for sulfide minerals, as follows:

Agent	Degrees
Zinc and cetyl xanthate	89
Copper and benzyl xanthate	72
Copper and amyl xanthate	88

These figures are suggestive and further work is required.

EFFECT OF CONCENTRATION OF XANTHATE

Table 6 shows that the concentration of the xanthate has little effect on contact angle provided that it is somewhat above the critical value at which contact occurs. It is suspected that, given sufficient time and provided that there are no disturbing factors, the full ethyl xanthate angle (corresponding to a complete xanthate film) would be obtained in every case where contact occurs.

The high angle of 80° in the most concentrated solution was due to the ormation of dixanthogen, droplets of which were visible.

TABLE 6.—*Dependence of Contact Angle on Concentrations*

A Freshly Ground Galena Surface Was Prepared for Each Concentration

Concentration, Mg. per Liter	Contact Angle, Deg.
½ KEtX	Nil up to 16 hr.
1	29 at 1 hr.; 40 at 16 hr.
2½	50 at 1 hr.
5	53 at 40 min.
20	58 at 40 min.
125 KEtX or NaEtX	58 at 40 min.
200 NaEtX	60 at 40 min.
350	59 at 40 min.
570	61 at 40 min.
2750	60 at 24 hr.
	80 after 3 days (cloudy solution)
	80 after 6 days.

The choice of concentrations of the reagents to be used in experiments of this type is difficult. To arrive at a satisfactory correlation between experiments with single crystals and plant-scale flotation operations it is essential to distinguish between consumption of reagents and concentration of reagents. It is customary for operators to specify additions in terms of pounds of reagents per ton of ore treated. When the pulp density (ratio of solids to water) is known, the original concentration of the reagent follows. Consumption of reagents, however, generally reduces this concentration. The final concentration must be sufficient to cause the desired action. This final concentration is that which should be used in these bubble tests (in which consumption of reagents is usually negligible). Generally, however, the final plant concentration is not known, and some arbitrary connection between small scale and plant scale must be adopted. Fortunately, as shown by Table 6, a reasonable excess of xanthate has little effect. It is advisable, therefore, to have an excess, and generally the concentrations adopted for all reagents have approximately corresponded to the actual additions in practice. One pound of reagent per ton of ore corresponds to an initial concentration of 150 mg. per liter for a pulp containing 25 per cent solids, to 225 mg. per liter for 33 per cent solids, and to 450 mg. per liter for 50 per cent solids, and so forth.

Rate of Development of Contact Angle

The preceding tables have contained some information on the rate of development of contact angle. The equilibrium condition is usually closely approached within 10 min.; however, 30 min. is the average time allowed for the attainment of that equilibrium, although occasionally a longer period, up to 2 hr., is required before the angle becomes constant.

Contact with the bubble is generally easily effected. As soon as the air bubble touches the surface it spreads out. On occasions, particularly when the surface is approaching a condition of nonsticking, considerable distortion of the bubble is necessary before contact occurs, even though the equilibrium angle may be normal. The presence of alkalis is often responsible for slowness in sticking. With a highly polished surface the same difficulty may be encountered. In the former case, it is possible that the condition of the bubble and not that of the surface is responsible, for since alkalis are weak frothing agents, it follows that the presence of alkali has some influence on the bubble wall. In the second case the cause may be the lack of a nucleus at which coalescence may commence. It has been suggested that contact never can be effected unless there is a nucleus of air about which it may begin. This might well be, but it appears impossible by direct observation either to confirm or to disprove this. It is, moreover, difficult to explain the formation of the original nucleus. It is interesting to consider the conditions of both the surface and the bubble prior to contact. The mineral is surrounded by an oriented layer of xanthate molecules, probably with the ethyl groups outward. The surface is probably electrically charged. The air bubble is also surrounded by an oriented layer of hydroxyl and/or xanthate ions. It also is probably charged. It is known that if the air itself can come into contact with this layer of ethyl groups contact will occur, provided that certain relations are satisfied by the three interfacial tensions. But if the air is protected by an adsorbed layer of hydroxyl or xanthate ions, how can contact occur? The problem is further complicated by the irregularities of the surface of the mineral. Moreover, water must be squeezed out. It is apparent that distortion of the bubble, with a consequent generation of a new air surface, will be conducive to contact between the air and the conditioned mineral surface. In a flotation cell where contacts between the bubble and mineral cannot endure for more than a fraction of a second it is apparent that violence of impact will help considerably, for it will tend both to create a new air surface and to squeeze out water. The latter process, it would seem, would be greatly facilitated at irregularities in the solid surface. Here one might digress to point out that though thermodynamics provides a criterion as to whether sticking between air and mineral is possible (see later) it is powerless to predict the rate at which sticking will occur. That is a point which can only be determined experimentally.

Another time effect is interesting: A polished galena surface becomes inert if left in contact with distilled water for many hours. It is then apparently unresponsive to xanthate. Wiping on linen will restore its activity, but no chemical treatment has been found effective. The reduced recovery in a pulp which has been left standing between grinding and flotation may be due to this condition, but it is possible that the violent agitation of the flotation machine would be as effective as wiping by linen in removing it. It is curious that, although the inert surface when treated by xanthate will not stick to an air bubble, it is nevertheless affected by the xanthate. For if it is removed from the solution, washed in several changes of water and then after wiping with linen is tested again without further xanthate treatment, the customary angle of contact develops. This test and many similar ones have demonstrated the extraordinary permanency of the xanthate effect.

Use of Activators for Sphalerite and Other Minerals

In order that our work on the action of activators may be presented rationally, our conclusions will be stated first; the experimental work which is then described is offered in support. To obtain an oriented coating of xanthate on the surface of any solid it appears to be necessary that the metallic constituent or ion of the atomic lattice of which the solid is built should form with the xanthate a salt which is relatively insoluble in the water phase.[3] Thus, if a copper mineral is to be floated by ethyl xanthate it is necessary that copper xanthate be insoluble in the solution in the cell. This is a deduction from experiment. It is to be emphasized that unless the nature of the adsorption of xanthates were definitely known to be purely chemical no such deduction would be possible on theoretical grounds. If experiment shows that this generalization is invariably satisfactory, it may be deduced that the xanthate adsorption is of a chemical nature.

The ethyl xanthate of zinc is relatively soluble. Zinc minerals cannot therefore be floated by ethyl xanthate alone. The solubility of the salts of the xanthates decreases rapidly, however, as the size of the alkyl radicle increases. Nevertheless, zinc amyl xanthate is so soluble that under ordinary conditions sphalerite is not floatable by potassium amyl xanthate. It is a well-known consequence of the law of mass action that precipitation of a salt may be aided by an increase in concentration of either of the constituent radicles. Applying this principle to the solubility of zinc amyl xanthate one finds that increases in zinc-ion concentration (by addition of zinc sulfate) or of amyl xanthate ion both result in the precipitation of zinc amyl xanthate, and the customary amyl-xanthate contact angle develops as soon as precipitation occurs. The concentrations necessary are discussed below. Similar figures are given for benzyl xanthate.

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[3] There is a distinction to be drawn between the solubility of the thin coating (probably monomolecular) of, say, copper xanthate and the solubility of copper xanthate in bulk. It cannot be assumed that they are identical, though we are now reasonably certain that they are closely connected. It is, of course, the former which must be used in this connection.

Chalcopyrite fails to respond immediately to methyl xanthate. Addition of one milligram of copper per liter (as $CuSO_4$) immediately results in contact. Bornite, which oxidizes more rapidly than chalcopyrite, responds more quickly to methyl xanthate. One can only conclude that a certain minimum concentration of copper is necessary in solution, in order that the solubility product for copper methyl xanthate may be exceeded. The number of instances of this kind could be multiplied considerably by including reagents other than xanthates.

The difficulties cannot be overcome in this way for sphalerite and ethyl xanthate. Only at a high concentration both of xanthate and of zinc sulfate is a precipitate formed. The high zinc concentration inhibits contact (see later). Use of one of the recognized activators, $CuSO_4$, for example, is therefore necessary with all concentrations of ethyl xanthate.

Certain tests in which sphalerite was subjected consecutively to the action of copper sulfate, potassium xanthate and cyanide in various orders are illuminating with respect to the nature of the activation by copper sulfate. Treating the surface first with a 20 mg. per liter solution of copper sulfate and then, after washing, placing it in a 25 mg. per liter xanthate solution, an angle of 60° developed. Treating it first with the xanthate solution and, after washing, placing it in 20 mg. per liter $CuSO_4.5H_2O$ solution, no angle of contact developed.

It is concluded that the sphalerite surface becomes coated by a permanent film of some copper compound when it is placed in a copper-bearing solution and that this coated surface subsequently reacts with xanthate. This same order of events is presumably followed if the sphalerite is placed in a solution containing both copper and xanthate.

On the other hand, if no copper is present and the surface is not already coated by copper, the placing of a sphalerite specimen in xanthate solution does not result in formation of a film that is subsequently responsive to copper. Further confirmation of these conclusions has been obtained by the action of cyanide. (See page 222.)

Sphalerite and Amyl Xanthate.—At a sphalerite surface in a solution of 9 mg. per liter $ZnSO_4.7H_2O$ to which was added amyl xanthate to give a concentration of 25 mg. per liter, a contact angle developed within 1 min. and at 20 min. the angle was 70° rising slowly to 88°. With 50 mg. per liter of the zinc salt and 125 mg. per liter of the xanthate the rate of development of the angle was faster. At these concentrations, a white precipitate of zinc amyl xanthate is formed.

Sphalerite and Benzyl Xanthate.—With 1 g. per liter K benzyl xanthate, between 16 and 32 mg. per liter $ZnSO_4.7H_2O$ is necessary to give a precipitate of the zinc xanthate, and a contact angle develops immediately at a sphalerite surface in a solution at the latter but not at the former concentrations. The solubility product of zinc benzyl xanthate, $[Zn^{++}] \times [\text{Benz } X^-]^2$, is approximately 9×10^{-10}, corresponding to a concentration of 0.26 g. per liter for a saturated solution. When the actual concentrations of the ions are such that 9×10^{-10} is exceeded, precipitation occurs and if also the xanthate is in stoichiometric excess of the zinc concentration contact will occur.

Activators Other than Copper Sulfate.—Tests are reported in Table 2 which show that mercuric chloride and silver sulfate are effective activators for blende. It may be inferred that sphalerite is, in these cases, coated with a film of mercury or silver sulfide and that the response to xanthate is due to the inherent floatability of these sulfides by ethyl xanthate. Mercuric and silver xanthates are both much less soluble than the corresponding zinc salts. This probably accounts for the contact between air and a sphalerite surface previously modified with salts of these metals. Mercuric and silver sulfides are both less soluble than zinc sulfide, which accounts for the activation of sphalerite by mercuric and silver salts.

As lead sulfide is less soluble than zinc sulfide, and further, since lead sulfide is coated by xanthate without addition of any other agents, one might expect that lead salts would activate sphalerite. Experiments proved that they do so, provided that the precipitated lead xanthate is not allowed to settle on the sphalerite surface. It is better for the sphalerite to have contact with the lead salt before the xanthate than to have these contacts in the reverse order. The angle develops faster and approaches more nearly the angle characteristic of ethyl xanthate. With 10 mg. per liter $Pb(NO_3)_2$ and 25 mg. per liter KEtX, the angle of contact was 57° within 5 min. and remained unaltered for over an hour. Adding the same reagents in the reverse order (18 mg. per liter of the lead salt, however) the angle of contact after 15 min. was uneven and lower. After contact for one hour and wiping on linen to free the surface from the precipitated lead xanthate, an angle of 54° was attained.

To evaluate the practical significance of this test, it was repeated in sodium carbonate solution. It was anticipated that most of the lead would be removed as carbonate and that insufficient would remain in solution to activate the blende. Two tests resulted in a striking confirmation of this view. When the carbonate was in stoichiometric excess no contact could be induced; the pH value in the actual test was 7.9, owing to the carbonate. With the lead in excess, contact was easily effected, even though sufficient caustic soda had been added to raise the pH value to 8.1.

The effect of excess of a silver or mercuric salt is interesting, especially when compared with that of excess copper. An excess of the former is harmful, whereas excess of copper sulfate is certainly not very harmful, if at all injurious. These effects are exhibited in Table 7. Excesses of heavy metal ions have been studied by Gaudin and collaborators(22) and by Taylor and Bull(10) in direct flotation experiments. Bartsch(6) has shown that they have an influence on the froth.

TABLE 7.—*Effect of Excess of Metallic Ions on Contact Angles at Sphalerite Surfaces*

Successive Additions	Solution Composition After Addition		Angle of Contact		Stoichiometric Excess of
Ag_2SO_4 series	Ag_2SO_4	KEtX, mg. per l.	Swansea Blende	Joplin Blende	
25 mg. per l. KEtX........	Nil	25	Nil	Nil	Xanthate (No Ag present)
50 mg. per l. Ag_2SO_4.......	50	25	Nil	Nil	Silver
25 mg. per l. KEtX........	50	50	57	58	Xanthate
50 mg. per l. Ag_2SO_4.......	100	50	Nil	Nil	Silver
$HgCl_2$ Series	$HgCl_2$	KEtX	Swansea	Joplin	Excess of
25 mg. per l. KEtX........	Nil	25	Nil	Nil	Xanthate (No Hg present)
15 mg. per l. $HgCl_2$........	15	25	59	52	Xanthate
20 mg. per l. $HgCl_2$........	35	25	Nil to slight		Mercury
25 mg. per l. KEtX........	35	50	57	50	Xanthate

Fig. 6 summarizes the position with respect to the necessity for adding activators when using various alkyl xanthates. This graph, of course, applies only for dilute xanthate solutions (less than 100 mg. per liter) and the black line must follow, roughly, a curve of equal solubility. Of greater value would be a determination of the solubility product of each of the metallic xanthates and of the metallic-ion concentration-time curve (Zn^{++}, Pb^{++}, etc.) for each mineral in distilled water. When the concentration of the xanthate is known it would then be possible to calculate whether a precipitate would be formed in any given pulp, and thence, probably, whether contact would be possible. The possibility of controlling selective flotation by metallic-ion concentrations is attractive. Indeed, the extensive use of alkalis and cyanides in flotation is probably due to their actions in this way.

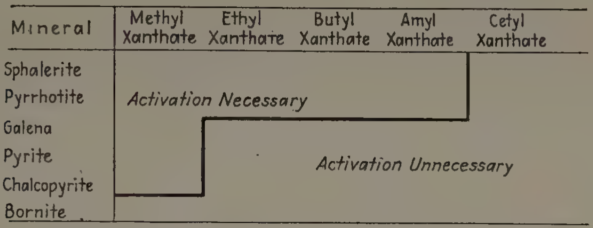

FIG. 6.—ACTIVATORS WITH VARIOUS XANTHATES.

ETHYL DIXANTHOGEN AS A COLLECTOR

Repeated reference has been made to the influence of the dixanthogens on contact angle. The evidence obtained relating to their effects is now presented:

In studying the action of concentrated solutions of potassium ethyl xanthate on galena it was observed that the concentration at which the contact angle changed from 60° to 80° was different for samples from two different makers. It was deduced that the increase was due to an impurity. Purification by recrystallization from alcohol of the less pure sample resulted in a decreased tendency to develop the 80° angle, though it did not completely eliminate this tendency. It was also observed that the occurrence of the high angle coincided with the appearance of a milkiness in the solution. An ether extraction cleared the solution. After the ether layer was removed and the dissolved ether had evaporated, the angle of contact was 60°. Moreover, an ether extraction of the impure xanthate recovered an oil of which an emulsion gave an angle of 77° at a galena surface.

Similar results were obtained with copper minerals. A specimen of chalcocite showed an angle of contact of 80° in potassium ethyl xanthate solution containing 125 mg. per liter. When the latter was shaken with ether and equilibrium reestablished in the presence of a layer of ether, the angle[4] had fallen to 54°. The ether was removed and the excess evaporated, and the angle rose to 79°. A second ether extraction lowered the angle to 53°.

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[4] It was shown in an independent experiment that a supernatant layer of ether lowered the ethyl xanthate angle from 60° to 51°.

Since it was suspected that the impurity responsible for the 80° angle of contact was ethyl dixanthogen, this compound was tested. Prepared by the action of iodine on potassium ethyl xanthate in aqueous solution, it may be extracted by ether, from which it crystallizes as a pale yellow solid on evaporation of the ether:

$$2\text{EtO.CS.SK} + \text{I}_2 = \text{EtO.CS.SS.CS.OEt} + 2\text{KI}$$

The compound is relatively insoluble in water and two methods of obtaining a saturated solution were used: In the first the solid was shaken with water; in the second a few drops of an ether solution were added to water. The former resulted in a suspension of the solid, the latter in an emulsion of the supercooled liquid in water. It is in the latter condition that the compound is produced by the spontaneous decomposition of a strong aqueous solution of potassium ethyl xanthate; this might be expected because of its low melting point.

Chalcopyrite gives different results with these two solutions. The emulsion coats it with a thin adherent liquid film on which the angle of contact is 80° to 81°. The suspension of the solid, which obviously cannot spread, reacts more slowly, and the maximum angle attained, 60°, suggests that some of the dixanthogen may be reconverted to xanthate before or during adsorption.[5]

It would appear, therefore, that the higher of the two characteristic angles obtained in a xanthate solution is due to the appropriate dixanthogen; that this compound is often a constituent of commercial samples; that it is formed spontaneously by oxidation of the xanthate; and that the presence of copper salts greatly facilitates its formation because cupric ion is an oxidizing agent. It is equally apparent that the success of the dixanthogens as collectors is due to their ability to "oil" certain mineral surfaces.

Dixanthogen is not selective between sulfide minerals, but displays some selective action in that it does not oil silica or an oxidized mineral such as anglesite. Since it is so readily formed by oxidation of xanthates, it seems likely that it may have an influence in flotation by xanthates, especially after long aeration. There has been no opportunity to examine the effects of alkalis, cyanide, and other compounds on the spreading of dixanthogen over the surfaces of sulfide minerals; the subject is one of outstanding interest, however, wherever xanthates are used.

CARBON BISULFIDE AS A COLLECTOR

Kellerman and Bender(23) state that xanthates decompose so rapidly into carbon bisulfide and alcohols that any effect of xanthate as such is impossible. The better floatability obtained upon addition of xanthate is, they claim, due to the presence of carbon bisulfide, which acts as a collector, and of the alcohol (ethyl from ethyl xanthate and so on), which acts as a frother. Since the experimental data upon which the hypothesis was based was not convincing, it was considered desirable to obtain more conclusive evidence than those authors had presented.

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[5] This indicates that there is probably an equilibrium mixture of xanthate ions and dixanthogen in such solutions, which could naturally be obtained by starting with either compound. The dixanthogen, if liquid, is dominant with regard to adsorption, for on addition of dixanthogen to a xanthate solution the contact angle rises from 60° to 80°.

Solutions containing 25 mg. per liter and 700 mg. per liter respectively of carbon bisulfide had no measurable effect on polished galena surfaces. These concentrations are higher than the amounts that could be produced by hydrolysis of the usual additions of xanthate. As the bisulfide solution was acid ($pH = 3.4$), the pH value was adjusted to 6.6, but this made no difference. When, however, the solution was supersaturated with respect to carbon bisulfide (over 2 g. per liter) sticking could be induced; the surface was coated with a visible film of carbon bisulfide. Sticking under these conditions probably accounts for the authors' success in small-scale tests with carbon bisulfide as a flotation agent. No concentrations were measured by them, and there may have been opportunity for the particles to become "oiled" with carbon bisulfide.

Different angles of contact are obtained with different xanthates; this is further evidence that Kellermann and Bender's claim is unjustified. Again, in strongly acid solutions ($pH = 1$) ethyl xanthate decomposes rapidly; contact is then impossible, despite the production of carbon bisulfide.

EFFECT OF ALKALIS ON CONTACT ANGLE

Though the present study of the action of alkalis on the contact between minerals and air is incomplete, the subject is of such importance that the results obtained up to the present may prove of interest. A complete solution would demand a knowledge of the effect of alkalis at each of three interfaces—air-water, water-mineral, mineral-air. Because interfacial tensions at solid surfaces cannot be measured, it is impossible to obtain this knowledge directly and an attempt must be made to determine the nature of the changes at the mineral surface by inference. Use of bubbles of organic liquids instead of air may prove useful in this connection; their application is considered below.

Galena.—With galena, the time effect noted on page 211 has been observed. Alkalis at pH values above 8 introduce considerable difficulty in effecting contact. Greater distortion of the bubble is necessary and longer apparent contact is required before real contact occurs. The cleaning of the surface by wiping with linen lessens but does not eliminate this difficulty, which seems to be partly attributable to the condition of the bubble itself. The characteristic angle of contact is not obtained without wiping but, on wiping, the full angle of contact is always obtained up to $pH = 10.1$. By $pH = 10.3$ contact has become altogether impossible in a 25 mg. per liter KEtX solution. The change from a 60° angle to a zero angle of contact is thus sudden at a clean surface. It is more gradual, however, if the surface is not cleaned by wiping, but the point at which contact ceases to be possible is unchanged.

Table 8 shows how the xanthate concentration affects this critical pH value, above which contact is impossible and below which full contact is possible. The determinations are probably correct within 0.2 unit.

TABLE 8.—*Effect of KEtX Concentration on Critical pH Value.*

Concentration of KEtX, Mg. per Liter	Critical pH Value
500	11.9
100	11.0
25	10.2
5	9.1

These critical pH values are independent of the alkali used. Lime, sodium carbonate and caustic soda seem equally effective as depressors. That the carbonate and calcium ions have little influence on the critical value was proved in an independent series of tests in which 1½ g. per liter of CO_3^{--} and 350 mg. per liter Ca^{++} respectively were present. All the other tests, however, have been in sodium carbonate solution of concentration 75 to 250 mg. per liter with additions of hydrochloric acid or caustic soda, where necessary, to adjust the pH to the desired value.

The alkali effect is not as permanent as the xanthate effect. A galena surface already conditioned by xanthate, when washed and placed in distilled water, will retain air avidity corresponding to the usual angle of 60°. Gradually this avidity is lost as the xanthate film dissolves. On the other hand, a galena specimen of which the surface has been prepared in a 1 g. per liter Na_2CO_3 solution is not permanently affected thereby, for on washing and placing in xanthate solution the customary contact angle immediately develops.

Alkalis are not so effective in preventing contact with air in an amyl xanthate solution. With a concentration of 25 mg. KAmX per liter the critical value is 11.8. A curve showing critical pH value plotted against concentration of xanthate could therefore be obtained for each xanthate, but the curves for consecutive homologues would not lie far apart.

Acids rapidly destroy the contact in xanthate solutions, provided that the pH value is approaching unity. It is known that acids decompose the xanthates, the reaction for ethyl xanthate being

$$KS.CS.OEt + HCl = KCl + CS_2 + EtOH$$

After a few minutes contact between N/10 acid and ethyl xanthate solution, no xanthate can be detected by the copper sulfate tests, and subsequent addition of alkali is unable to regenerate the xanthate.

Tests in which a drop of benzene or other organic liquid replaced the air bubble were undertaken in an endeavor to determine whether the mineral surface undergoes any abrupt change at the critical value. It would be deduced that, if a bubble of air and drops of several organic liquids all cease to make contact with a galena surface at the same pH value, the surface of the mineral undergoes some marked change at this point. Unfortunately, all the liquids tried do not exhibit identical critical pH values in 25 mg. per liter KEtX solution. The significance of the differences is not apparent.

In the absence of xanthate, benzene[6] sticks to a galena surface up to a critical pH value of 8.2; the corresponding value for carbon tetrachloride is between 10.0 and 9.6. It may be deduced that the surface condition of the drop is of importance in determining whether contact shall be possible, and that the condition of the mineral surface is not wholly responsible. It is not then so surprising that the critical values in a 25 mg. per liter solution of ethyl xanthate should be 10.2 and 11 for air bubble and carbon tetrachloride drop respectively. It is, however, impossible to decide at what pH value the xanthate film leaves the surface. That it ultimately does leave the surface is certain, for lead xanthate dissolves rapidly in solutions of strong alkalis at pH values greater than 12. No doubt the solubility is due to the formation of plumbates. Whether the action of alkalis on galena can be interpreted in terms of this action is uncertain. The tests with organic liquids suggest that air-mineral contact ceases to be possible while the xanthate film is still on the surface. If so, it is difficult to account for the different critical values given by different xanthates and for the variation of the critical value with concentration of xanthate. One might suggest tentatively that, prior to its complete removal, the xanthate film changes to basic xanthate, which is not air avid but which may permit the formation of contact angles with benzene and carbon tetrachloride bubbles. The tests with an air bubble possess the greater significance for flotation as customarily carried out.

Pyrite.—Using an alizarin yellow GG color disk for the estimation of pH value [7] it was ascertained that the limiting value above which sticking becomes impossible is 10.5. At this value, the surface of the pyrite had to be cleaned by linen to effect contact and the angle was then not quite 50°. Below $pH = 10$ contact is readily effected and at $pH = 9$ contact is rapid and the angle reaches 60°.

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[6] The contact angles with benzene are 55° and 110° in distilled water and xanthate solution respectively. Hysteresis is troublesome with all these organic liquids; angles of contact can only be estimated approximately.

[7] Some of the disks sold by a well-known German maker are not reliable in solutions such as are used in flotation. In identical solutions differences between disks for different indicators may amount to 0.6 unit. This disk was calibrated by the potentiometric method; it required no correction.

When using lime at $pH > 10.5$ (250 mg. per liter CaO), contact between air and pyrite in a 25 mg. per liter ethyl xanthate solution is not obtained, but contact between benzene and pyrite is obtained. The angle of contact is close to that for benzene and pyrite in xanthate-free solution, namely, 80° to 90°, which is well below that obtained at a xanthate-coated surface, namely, 110°. It is easy to distinguish between the type of contact at these two angles, though they cannot be measured accurately because of a large hysteresis effect.

It would be deduced that at about $pH = 10.5$, the xanthate film leaves the surface. It is suggested that the effect of the alkalis is to reduce the ionic concentration of the iron to such a low value that the iron xanthate film can no longer exist. Such a suggestion is little more than a conjecture; our excuse for offering it is that it points to the necessity for electrochemical determination of ionic concentrations.

Tests with amyl xanthate also indicate that the film leaves the surface. The contact angles for pyrite coated with amyl xanthate are 85° for an air bubble and up to 140° for a benzene bubble. It is easy to distinguish between a clean (xanthate-free) pyrite surface for which the contact angle is 80° to 90° and an amyl xanthate coated surface (140°). While 250 mg. CaO per liter prevents contact with an air bubble in a 25-mg. KAmX solution, it reduces the benzene angle of contact from 140° to only 80° to 90°. Acidifying the solutions leads to a recovery of the angle to 140°.

Sphalerite.—As no contact is possible in a 25 mg. per liter KEtX solution in the absence of activators, the depression of sphalerite must be studied in the presence of a heavy metal salt; $CuSO_4$, for example. It is true that in practice most of the copper is precipitated as hydroxide or basic carbonate,(24) but evidently the concentration of the copper ion in equilibrium with this is greater than that necessary to coat sphalerite. Physicochemical principles indicate that the actual copper-ion concentration—not the total dissolved copper—is the deciding factor for coating the sphalerite.

In conducting some preliminary tests, the copper sulfate additions of 10 mg. per liter were made to alkaline solutions of the desired pH value; the sphalerite was then conditioned for 30 min., and finally 25 mg. per liter KEtX was added. In solutions of pH value higher than 12, contact was often difficult or impossible to effect. On other occasions contact was immediate and the full angle was obtained up to $pH = 13$. Measurements of copper-ion concentration might provide an explanation of the inconsistencies between apparently identical tests. It is to be remembered that the pH values of flotation circuit liquors are invariably well below 12.

Alkalis are not nearly as effective in preventing the activation of sphalerite in the presence of copper as they are in depressing pyrite and galena in its absence. Moreover, the behavior of activated sphalerite with respect to alkalis is very similar to that of the copper minerals. Gaudin(25) has stated, "A somewhat higher alkalinity is desirable in zinc circuits of complex lead-zinc ores than in the lead or iron circuits of the same."

Effect of Cyanide on Contact Angle

Because cyanides are almost universally used in flotation plants, a detailed study of their action on contact angles at the surfaces of the sulfide minerals is desirable. Only a preliminary survey with three of the minerals has yet been made in this laboratory. The effects of cyanide on the galena and pyrite contact angles have been determined over a fairly wide range, and those on activated sphalerite have been determined over a limited range.

No systematic attempt has yet been concluded which would explain the modus operandi of the cyanide effect on pyrite. An early distinction is necessary, however, between the functions of cyanide in depressing activated sphalerite and in depressing unactivated pyrite.

There is no doubt that generally the depression of sphalerite is due to the formation of complex cuprocyanides and is therefore a de-activation rather than a depression. Nevertheless, there are two conceivable modes of action by which the conditioning might be prevented:

1. De-activation: The copper-ion concentration might be reduced so low that sphalerite would not be coated with cupric sulfide.[8]

2. Depression: Though the sphalerite may be coated, the copper-ion concentration might be so low that no copper xanthate forms on the surface. Under certain conditions, chalcopyrite itself may be depressed by cyanide.

Physicochemical principles indicate that the ionic concentrations of copper in equilibrium with solid copper xanthate, copper and zinc sulfides, and with the complex cuprocyanide ions are the determining factors for the course of the reaction. In any given case the zinc-ion and the xanthate-ion concentrations would also have to be known before the course of the reaction could be predicted. The complete solution of the problem thus resolves itself into a complex series of determinations of ionic concentrations.

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[8] Gaudin, Haynes, and Haas(21) have summarized the evidence in favor of the view that this sulfide is formed at the surface. The argument is unchanged, however, if it is assumed that cuprous sulfide is present on the surface.

Nothing can be said with certainty concerning the reasons for the depression of pyrite by alkaline cyanide solutions. Our conjecture is that here, again, it is a question of the ionic concentrations—in this case of the iron and the xanthate. It is well known that iron forms complex cyanides which are in equilibrium with a very low ionic concentration of iron. Presumably, where cyanide has been effective as a depressant, the ionic concentration of the iron is below the minimum value for xanthate formation. How the alkali influences results is again a matter for conjecture. Possibly the cyanide-isocyanide equilibrium is influenced by alkalis, or possibly hydroxy-cyanide or carbonate-cyanide complexes of the Werner type are formed. It is significant that lead forms no complex cyanide nor is the contact at a galena surface affected by cyanide.

Galena.—Even in solutions of which the pH value is approaching the critical value, 10.2, sodium cyanide up to 1000 mg. per liter has no effect on contact angle provided that the surface be wiped by linen to clean it.

Pyrite.—Table 9 summarizes the results for pyrite. The xanthate concentration having been kept constant at 25 mg. per liter KEtX, the critical pH value above which contact between air and mineral ceased to be possible has been determined for a number of concentrations of cyanide.

TABLE 9.—*Effect of Alkaline Cyanide on Contact Angles at Pyrite Surfaces*

Sodium Cyanide Concentration, Mg. per Liter	Critical pH Value (Approximate)
Nil	10.5
1	8
10	7.5
100	7
500	Not less than 6.6

Sphalerite.—Tests with sphalerite are complicated by the presence of an activator. The concentrations of cyanide, of xanthate, of the activator and the pH value are variables, which obviously should be investigated. Unfortunately, the pH value alone does not specify the condition of the copper. Pickering(24) has demonstrated that the relative amounts of carbonate and bicarbonate influence the proportion of the copper in positive and negative (cuprocarbonate) ions. They also have an obvious influence on pH value itself. A complete study of the influence of all these variables is therefore necessary before the effects of cyanide can be clearly defined.

Simplification is necessary. Some of the variables must be kept constant while the effects of others are determined. The plan followed has been to fix the xanthate concentration at 25 mg. per liter and to determine the minimum cyanide additions for depression corresponding to various copper sulfate additions at a series of fixed sodium carbonate concentrations. Unfortunately, the cyanide additions influence the pH value, and the absorption of CO_2 from the air affects both the pH value and the carbonate-bicarbonate ratio. The maintenance of any desired condition is therefore difficult. Though the work is not sufficiently advanced for detailed presentation, one or two generalizations stand out clearly:

On allowing a sphalerite specimen 30 min. contact with solutions containing various combinations of cyanide, sodium carbonate and copper sulfate, and then adding the xanthate, it is almost invariably found that sticking occurs only if there is a precipitation of the yellow copper xanthate on addition of the xanthate.

The results from some preliminary experiments show that: (*a*) In approximately neutral solutions, between 10 and 15 mg. per liter of NaCN counteracts the influence of 10 mg. per liter $CuSO_4.5H_2O$; and more than 125 mg. per liter of the cyanide is required for 150 mg. per liter of the copper salt. (*b*) As sodium carbonate is added and the pH value rises, 15 mg. per liter NaCN + 20 mg. per liter Na_2CO_3 are sufficient to overcome the effects of 25 mg. per liter of the copper salt at $pH = 9$; 10 mg. per liter NaCN + 50 mg. per liter Na_2CO_3 are equally effective at pH just below 10; but 5 mg. per liter NaCN + 500 mg. per liter Na_2CO_3 are ineffective at $pH = 10.7$.

More fundamental information can be obtained by studying the action of the variables when they are applied to the surface of the sphalerite consecutively, as in the series of tests described below:

A. Tests have already been described on page 212 which show that an initial treatment with copper sulfate followed by treatment with xanthate in copper-free water results in the normal contact angle.

B. Sphalerite was activated in 25 mg. per liter $CuSO_4.5H_2O$ solution for 15 min., washed for 1 min. in running water, placed in 150 mg. per liter NaCN solution for 30 min., again washed, and finally placed in 25 mg. per liter KEtX solution. No contact was possible. This test proves that cyanide can remove a film containing copper from a sphalerite surface.

C. Sphalerite was placed for 30 min. in a solution containing 150 mg. per liter NaCN. After washing it was conditioned by 10 mg. per liter $CuSO_4.5H_2O$ and then gave the usual contact with xanthate (61°). This proves that cyanide has no permanent effect on the surface of sphalerite.

Determinations of the influence of cyanide on the air-mineral contact at a sphalerite surface in cetyl xanthate solution would be interesting, for no activator would then be necessary.

INFLUENCE OF NATURE OF GAS ON CONTACT ANGLE

Adams(26) has stated that the floatability of minerals is dependent on the gas used. This claim is at variance with the results obtained by us with solutions of xanthate, as is evidenced by the data of Table 10.

TABLE 10.—*Effect of Nature of Gas on Contact Angle*

Gas	Angle of Contact, Deg.
Air	60
Oxygen	62
Hydrogen	60
Nitrogen	59
CO_2	61
SO_2	50 ± 5 (pH < 3)

The value for SO_2 may have been influenced by the high acidity of the solution causing decomposition of the xanthate before the measurements were completed. Because of the solubility of the gases, the angles for SO_2 and CO_2 were difficult to measure.

It is perhaps surprising that the different gases do not produce some difference in the angle of contact, which is determined by the values of three interfacial tensions. Of these, that of the liquid-gas interface is known not to depend greatly upon the nature of the gas with which the liquid is in contact. It is probable that the second interfacial tension—that between the galena and the liquid—will remain practically uniform, whatever the nature of the gas. The third interfacial tension—that between the solid and the gas itself—apparently also is unaffected by the nature of the gas. This is surprising. It is well known that adsorption of different gases on platinum depends to a large extent upon the nature of the gas and *a priori* one would have expected a similar relationship in the case of the galena. As a possible explanation of this apparent independence of the nature of the gas it might be assumed that sufficient air or oxygen was present in every case to enable the bubble to act as a dilute solution of, say, oxygen. Should this be the correct explanation, it is implied that a small concentration of oxygen in the bubble is as effective in determining the angle of contact as a small concentration of xanthate is in the solution. An elaborate series of experiments is necessary to determine whether this is the correct explanation and, as it could have no bearing on practical flotation, it does not seem necessary to continue experiments with these gases.

INFLUENCE OF SOME OTHER MODIFYING AGENTS

In addition to those already referred to, other modifying agents were investigated and the results follow:

Sodium Silicate.—This compound is used as a modifier in the differential flotation of lead-zinc ores. Sufficient work has been done to show that the angle of contact at galena and activated sphalerite surfaces is substantially unaffected by 500 mg. per liter, but it is not impossible that the pH range over which contact can be effected, the action of $CuSO_4$

and others, might be influenced by it. Or its effect in flotation may be due to its influence on the froth, for silicates readily pass into the colloidal state and colloids have a marked influence on frothing. (See Bartsch⁽⁶⁾ and Gaudin, Haynes and Haas.⁽²¹⁾)

Potassium Dichromate.—Bubble tests show that potassium dichromate is an effective depressant both for galena and activated sphalerite. The depression of the former is generally attributed to the formation of an insoluble chromate film on its surface. The tests with crocoite described earlier indicate that this explanation is inadequate. It is usually assumed that sphalerite will not be influenced because of the relatively high solubilities of zinc chromate and zinc dichromate. Gaudin, Haynes and Haas⁽²¹⁾ have shown, however, that the flotation of sphalerite is affected by dichromate.

GALENA.—The angle of contact at a galena surface in 25 mg. per liter xanthate solution was reduced in 10 min. from 58° to zero by 200 mg. per liter $K_2Cr_2O_7$. After longer contact a yellow deposit—presumably of lead chromate—appeared on the surface; 40 mg. per liter of the dichromate reduced the contact angle to zero within half an hour. Furthermore, the angle obtained in a 25 mg. per liter amyl xanthate solution was rapidly reduced to zero by 100 mg. per liter $K_2Cr_2O_7$.

SPHALERITE.—When added to a solution of xanthate containing a sphalerite specimen activated by $CuSO_4$, dichromate destroys the mineral-air contact. Thus, after 10-min. contact with such a sphalerite surface at which the angle was 59°; 50 mg. per liter $K_2Cr_2O_7$ reduced the angle to a small and variable value and after 45 min. to zero.

It was suspected that this might have been due to destruction of all the xanthate by dichromate. The sphalerite surface was therefore given contact with a 100 mg. per liter dichromate solution for 30 min. prior to being placed in contact with the usual reagents. Though this treatment was followed by 30-min. contact with 10 mg. per liter $CuSO_4.5H_2O$ solution which was then made up to 25 mg. per liter with respect to KEtX, no contact between mineral and air could be induced.

Zinc chromate and zinc dichromate are both so soluble that neither could possibly have been precipitated on the sphalerite surface. Yet the surface evidently has been altered. It is difficult to explain the effect of the chromate without recourse to hypotheses for which there is no evidence. Since it does not conform to the solubility hypothesis it is hoped that time may be found later to study it more closely.

Zinc Sulfate.—This reagent is widely used in practice as a depressant for sphalerite, especially in conjunction with sodium cyanide and alkali.

It has already been shown that, in carefully regulated amounts, zinc sulfate may often be used as an activator for sphalerite. Large quantities, however, especially if in stoichiometric excess of the xanthate, may function as a depressant. If copper sulfate has been used as the activator, however, zinc sulfate is not an effective depressant. A sphalerite specimen was placed in a solution containing 500 mg. per liter $ZnSO_4.7H_2O$ together with the usual addition of 10 mg. per liter $CuSO_4.5H_2O$. To the mixture was added 25 mg. per liter ethyl xanthate and after 30 min. an angle of contact of 61° had developed. This was practically unchanged (60°) by the addition of 2 g. per liter of the zinc salt. Again, 300 mg. per liter $ZnSO_4.7H_2O$ did not prevent activation by 2 mg. per liter of the copper salt—(that is, 0.5 mg. Cu per liter).

It is to be remembered in interpreting these results that zinc sulfate is generally used in conjunction with cyanide in an alkaline solution. Tests with zinc cyanide might prove instructive.

Zinc sulfate in concentrations up to 500 mg. per liter had no effect on the contact angle at a galena surface. When used in conjunction with cyanide, as in the following test, it was also without significant effect. A ground galena surface, placed in a solution containing 25 mg. per liter KEtX, 250 mg. per liter $ZnSO_4$ and 100 mg. per liter NaCN developed a contact angle of 55°, measured at 30 minutes.

Neutral Salts.—In determining the influence of alkalis and acids on the galena-xanthate angle of contact, lime and hydrochloric acid respectively were used. It became necessary to evaluate the effect of calcium and chloride ions. N/10 potassium chloride in the presence of 25 mg. per liter of xanthate gave the customary angle of 58°. Likewise, a solution containing 1 g. of calcium nitrate per liter had little effect on the contact angle. It is deduced that neutral salts, apart from those containing a heavy metal likely to form an insoluble xanthate or sulfide, are without effect upon the angle of contact.

Common Ions, for Example, Pb^{++} *and* S^{--} *on Galena.*—The sulfide ion acts as a depressant for galena—not a sensitive one—but at a concentration of 500 mg. per liter it was able slowly to destroy contact in a solution containing 25 mg. per liter KEtX. Galena placed in a solution containing both xanthate and hydrogen sulfide at the foregoing concentrations does not develop a contact angle.

In moderate concentration, lead acetate has no great effect upon the angle of contact at a galena surface conditioned by xanthate. At higher concentrations, however, it is a complete depressant; 7 g. per liter of acetate is insufficient to prevent contact in the presence of 25 mg. per liter of the xanthate after long immersion.

Lead xanthate is soluble in a moderately concentrated solution of lead acetate. The failure to develop contact becomes apparent only when lead xanthate is no longer precipitated from the solution by the xanthate, and is evidently due to the destruction of the xanthate ion as such.

A similar case has been studied by Talmud and Lubman,(27) who have found that the floatability of silver iodide by oleic acid is decreased considerably by a decided excess either of silver or of iodide ion.

EFFECT OF TEMPERATURE ON CONTACT ANGLE

Over a comparatively limited range it has been found that temperature has no effect on the value of the contact angle at a galena surface. Thus the angle of contact in a 25 mg. per liter KEtX solution at 50° C. was 58°. If the xanthate solution is maintained at 60° C. for ½ hr. this does not cause sufficient decomposition to affect the angle—which was again 58°. It may be concluded that the temperature coefficient is small, which is not surprising, because coefficients of surface tensions are usually small and it is upon these that contact angles depend.

Although the temperature coefficient of contact angle is small it does not follow that the range of contact with respect to pH variations, use of modifiers and other agents, is not influenced by the temperature. Indeed, if, as seems likely, contact depends on the solubility of metallic xanthate, the range of contact may be expected to vary with temperature. The rapidity with which equilibrium is reached would also be a function of the temperature.

EXPOSURE TO AIR

It is known that the flotative properties of ores which have been ground dry differ from those which are ground wet. There is some connection between this difference and the following tests.

Several galena surfaces prepared by grinding or polishing, and exposed overnight to the action of the air, have given angles of contact between 25° and 50° in distilled water. No xanthate was necessary. In every case, however, the angle decreased to zero on standing in water for several hours. Moreover, exposure to air prior to testing has no effect on the contact angle in potassium ethyl xanthate solution.

CONFIGURATION OF XANTHATE FILM

Sufficient evidence is submitted in the preceding sections to prove that, where a surface is rendered air-avid because of adsorption of ethyl xanthate, the angle of contact at the exterior surface presented by the adsorption complex is independent of the nature of the underlying solid, be it sulfide mineral, oxidized mineral, or even metal. There is available other evidence (not yet published) which suggests that this contact angle is independent of the nature of the adsorbed molecule, provided only that it contains the ethoxy or a similar group. What is the significance of this constancy? Surely it is that when ethyl xanthate is adsorbed on the surfaces of a number of solids the external surfaces are identical. The 60° angle of contact corresponds to the triple contact between air, water and xanthate molecules oriented in such a way that the ethyl group is outward. As far as we know, no such direct evidence has previously been advanced concerning the orientation of adsorbed films on solid surfaces. Orientation in films of the fatty acids on water, however, has been proved and a similar orientation has been tacitly assumed for solids by several writers on flotation.

It may be deduced that the space occupied by the ethyl group in the surface is independent of the nature of the solid surface. It is, in those few cases yet investigated, also independent of the nature of the anchoring polar group (xanthates and others). There seems to be only one way in which these conditions could be satisfied; namely, that as many xanthate molecules are adsorbed as the superficial area of the ethyl group will permit. Not only does the surface consist exclusively of ethyl groups, but these groups are packed as closely as possible.

The substitution of an amyl group for the ethyl group has marked effect on contact angle. Again, however, the nature of the exterior is apparently independent of the underlying solid. To date, the contact angles of 12 organic groups have been studied.

IMPORTANCE OF OIL FILMING

Both dixanthogen and carbon bisulfide are nonionizable, therefore the solubility or chemical theory which has been given so much attention by Gaudin and Taggart and their respective coworkers cannot be applied to them. Taggart, Taylor and Knoll(28) clearly recognize this.

On the basis of the present work it is necessary to distinguish between two types of surface change that will lead to sticking of an air bubble at the surface. It is not claimed that these two types embrace all collectors. (1) The surface is modified by adsorption of an oriented xanthate film on the surface as in flotation by potassium ethyl xanthate, and other soluble flotation agents. It has been claimed that the adsorption is of a chemical nature. (2) The surface is coated with a thin film of an insoluble oil, eucalyptus oil, dixanthogen and carbon bisulfide, for example.

NATURE OF ADSORPTION OF XANTHATES

Solubility or Chemical Theory.—When there is an adsorbed film of xanthate on the surface, the nature of the contact is determined by the alkyl or aryl group. Obviously, it is of prime importance that the factors which decide whether adsorption shall occur should also be discovered. After considering what others (Taggart, Taylor, and Knoll,(28) Gaudin, Haynes and Haas,(21) Taggart,(29) and Gaudin and Anderson(30)) have said, and the work of the present authors, we find that there is a strong connection between adsorption of xanthates and the solubility of the heavy metal xanthates, but we are not yet able to decide whether this amounts to an identity. Oil flotation definitely does not follow a similar course. Many cases have been cited which are apparent exceptions to the chemical theory of adsorption, and until these have been investigated, no further progress will be possible. Even though adsorption of xanthates were satisfactorily accounted for, there still remains unexplained the enhanced air avidity due to the adsorbed film. More has to be learned of the nature of the attractions across interfaces.

SUMMARY

The data presented in this paper are confined to experiments with the xanthate type of collector, although the range has been extended to other compounds, some of which, as far as we know, have not been used, or suggested for use, in flotation. Certain of our conclusions are naturally colored by the results obtained with these outside reagents. The more important conclusions are, briefly:

1. None of the common minerals experimented with, sulfides included, makes contact with an air bubble in distilled water, provided that the surface of the mineral is prepared by polishing in the way described. We believe that the mineral surfaces experimented with were clean and that they closely resembled the surfaces to be treated in a flotation machine. Whether they are chemically unchanged sulfide surfaces is another matter.
2. In a dilute solution of any xanthate, contact with air is easily obtained with some sulfide minerals. With other sulfide minerals and the lower xanthates, preliminary treatment with an activating agent (copper sulfate for sphalerite, for example) is necessary before contact is obtained. Even in the presence of activators, gangue minerals do not respond to xanthates.
3. The necessity or otherwise for the use of an activating agent runs parallel with the solubility of the xanthate compound with the metal in the mineral. For example, zinc ethyl xanthate is relatively soluble and no contact is obtained with unactivated sphalerite. Treatment of sphalerite with soluble salts of copper, mercury, lead or silver, which form less soluble xanthates, followed by immersion in ethyl xanthate solution renders the surface responsive to air. In conformity with the lesser solubility of zinc cetyl xanthate, it is possible to obtain contact in a cetyl xanthate solution without activators.
4. Whenever contact is obtained, the angle of contact is a constant for any particular xanthate, irrespective of the composition of the mineral. Side reactions in which we believe dixanthogen to be formed modify the angle with certain minerals and particular xanthates.
5. In the homologous series of xanthates, the contact angle increases with increasing number of carbon atoms in the nonpolar group of the compound. This increase, however, does not vary directly with the number of carbon atoms in such group; for example, the angle (measured across the solution) with methyl xanthate is 50°, with ethyl xanthate 60°, with amyl xanthate 86° and with cetyl xanthate 96°.

6. The angle obtained in an ethyl xanthate solution is not materially affected by increasing alkalinity until a certain "critical pH value" is reached, when contact ceases. This critical pH, above which contact is impossible and below which the full angle is obtained, differs for each mineral. It also varies with the xanthate concentration. If we may generalize from the single case of galena, it is fixed for any particular mineral and given xanthate concentration, irrespective of whether lime, soda ash or caustic soda is used.

7. The critical pH value is higher in an amyl xanthate than in an ethyl xanthate solution of the same concentration.

8. Alkalis and/or carbonates have an effect on the behavior of some activators. The efficacy of copper sulfate does not appear to be much affected by sodium carbonate solutions at high pH, but lead salts do not activate sphalerite in the presence of a stoichiometric excess of sodium carbonate. Caustic soda at pH = 8.1 does not destroy the activity of lead salts.

9. Cyanide in concentrations such as are used in practice is without effect on the contact between air and galena in ethyl xanthate solution; 250 mg. per liter does not alter the critical pH value.

10. With sphalerite, 10 to 18 mg. per liter of sodium cyanide counteracts the effect of 10 mg. per liter of $CuSO_4.5H_2O$, and 125 mg. per liter of sodium cyanide is enough to render innocuous 150 mg. of copper sulfate per liter. With increasing pH, cyanide becomes even more effective. There is adduced evidence which proves that sphalerite becomes coated by some copper compound in $CuSO_4$ solutions and that this may be removed by cyanide. We suspect that the formation of complex cuprocyanides is responsible for the effect of cyanide.

11. Cyanide lowers the critical pH value for pyrite. In the absence of cyanide this is approximately 10.5, but the presence of 1 mg. of sodium cyanide per liter lowers it to 8.5, and 100 mg. per liter to 7.

12. Air, oxygen, hydrogen, nitrogen and carbon dioxide give identical angles when brought into contact with xanthate-treated mineral. Sulfur dioxide lowers the angle slightly. Both SO_2 and CO_2 dissolve so readily that accurate measurement of the angle is difficult.

13. An air bubble can be attached to certain oxidized minerals in xanthate solutions, provided that fairly concentrated solutions of xanthate are used. With anglesite and cerussite no permanent contact is obtained in solutions containing 25 mg. per liter of ethyl xanthate but with 2 g. per liter the full characteristic angle for ethyl xanthate is obtained. Crocoite behaves in much the same way.

14. Metallic copper gives the same angle of contact with amyl xanthate solution as the sulfide minerals. Metallic zinc fails to respond to ethyl xanthate (even in the presence of copper sulfate), but gives the customary angle with cetyl xanthate.

15. Emulsions of carbon bisulfide and the dixanthogens render sulfide mineral surfaces responsive to air. Gangue minerals are unaffected by them.

16. Though results for collectors other than xanthates have not been included, the following result of outstanding interest may be mentioned here: Whenever contact between air and a mineral surface is effected in the presence of a solution of sodium diethyl dithiophosphate (sodium Aerofloat), the angle obtained is identical with that resulting from the use of ethyl xanthate. With sodium Aerofloat, however, it is necessary to activate both galena and sphalerite with copper sulfate.

17.[9] It is concluded that when a xanthate is adsorbed on the surface of any mineral the alkyl groups are oriented outward, and that they are packed as closely as possible.

18.[9] The significance of contact angle in flotation is discussed. A relationship has been deduced between the stability of the air-mineral contact on the one hand and the surface tension of the solution, the contact angle, and the area of contact on the other.

19.[9] It is concluded that hysteresis of contact angle is due to friction, and that it does not necessarily help flotation. With suitable precautions it may be eliminated in measuring contact angles.

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During the course of this research, the authors have received much help and advice from many members of the staffs of the companies associated with the work. Though it is impossible to mention each member individually, they desire to express thanks to all who have offered criticism and encouragement during its progress. Special indebtedness to Mr. Harry Hey has already been recorded.

The authors also thank Prof. E. J. Hartung for providing facilities for carrying out this investigation in the Department of Chemistry, University of Melbourne, and both him and other members of the University for help in obtaining supplies of chemicals and minerals, and for their part in many stimulating discussions of the problems.

It is difficult to assess one's indebtedness to each of the earlier contributors to the literature of flotation, but the authors have no hesitation in acknowledging the help of Professors A. M. Gaudin and A. F. Taggart and their collaborators.

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[9] This paper has been condensed. Reasons for these conclusions are in the original manuscript.

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DISCUSSION

(*Arthur F. Taggart presiding*)

R. Gahl,* Berkeley, Calif. (written discussion).—A short time ago I reviewed the book on flotation recently published by E. W. Mayer and H. Schranz.[10] In doing so I took exception to the authors' statement that the theory of contact angles had been abandoned because it failed to explain many flotation phenomena in modern practice. I added that actual experimentation with contact angles had proved extremely difficult and uncertain and that the testing of the theory by actual measurements had been unsuccessful and may perhaps be said to have been discarded.

When later I read the paper presented by Messrs. Wark and Cox, I gained the impression that the authors have succeeded in developing the technique of measuring contact angles to such a degree that it now appears quite satisfactory. Accordingly, the statement which I just quoted cannot be said to be correct any more and I wish to congratulate the authors on having accomplished this step, which is so important for the theoretical treatment of the flotation process.

A. M. Gaudin,† Butte, Mont. (written discussion).—From a scientific standpoint few papers on flotation have been published that come up to the high standard of excellence attained by the contribution of Messrs. Wark and Cox. This memoir is definitely pushing the realm of the unknown further into the distance, and indeed, marks a further step in the contribution to pure chemistry which the scientific phases of flotation are making.

Of the scientific discoveries presented by the authors, the most significant appear to be that: (1) The angle of contact at the mineral-gas-solution boundary produced by chemical collectors is the same for all minerals and for all collectors, provided the nonpolar radical in the collector is the same; (2) the angle of contact increases as the length of the hydrocarbon chain in the collector molecules increases; (3) the angle of contact is substantially the same for all collectors whose hydrocarbon chain is terminated by a phenyl or a phenyloid group.

As Messrs. Wark and Cox point out, the identity in contact angle for all sulfide minerals indicates that their surfaces, after coating with the reagent or its decomposition product, are identical. This is more direct proof of orientation of the coating with the nonpolar groups toward the solution than had heretofore been adduced.

The increase in contact angle with increase in length of hydrocarbon chain in the collector (Fig. 5) indicates increased shielding of the residual polarity of the mineral surface by the thickening layer of CH_2 groups. Correlation of the rate at which trigonometric functions of the contact angle change with the rate of change in thickness of the hydrocarbon coating should supply a valuable and suggestive insight into the nature and range of chemical forces. Such a correlation would consider, of course, that the collector coating is unimolecular—a consideration which is otherwise warranted.

The identity in contact angle obtained with sundry xanthates of the cyclic series indicates that the phenyl and other cyclic groups are not as completely nonpolar as aliphatic groups containing the same number of carbon atoms. This is in agreement with the conclusion derived by Langmuir, Harkins, Adam, and others from surface-energy relationships.

Elaboration of the authors' experiments to include studies of compounds other than the xanthates and dithiophosphates—as, for instance, of soaps, amines, thiophenols and mercaptans—ought to confirm further the conclusions enunciated above, and to indicate whether differences in the polarity of the polar part of the collector coatings are masked, or otherwise, by the shielding action of hydrocarbon chains of equal length.

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[10] *Eng. & Min. Jnl.* (1932) **133**, 49.

† Research Professor of Ore Dressing, Montana State School of Mines.

In many respects the results of operation and of practical testing appear at variance with the data of the authors. As is generally the case in situations such as these, both sets of data are correct, but some unwarranted assumption is made by some, or all, investigators, which if eliminated allows a reconciliation of apparently incompatible facts into a workable theory. In the following paragraphs an attempt will be made to effect just such a reconciliation.

In order to be able to ascertain the source of the apparent discrepancies, it is pertinent to estimate the extent to which conditions differ in practice and customary testing on the one hand, and the bubble-machine experiments of Wark and Cox on the other.

In the first place, the bubble-machine experiments deal with substantially plane mineral surfaces, whereas in flotation angular particles are found. This is acknowledged by the authors on page 210 under Rate of Development of Contact Angle. The mechanism of adherence of particles to bubbles in pneumatic machines, and probably also that in agitation machines, seems highly dependent upon the angularity of the particles.[11]

In the second place, it is by no means certain that the surfaces are identical in ball-mill-ground and in carborundum-wheel-ground mineral pieces. The authors take for granted that ball-mill grinding produces comminution by attrition, but recent studies of ball milling have rather favored the view that attrition plays a minor part. It may of course be argued, *a priori*, that the method of grinding should make no difference, and that may be so, but evidence should be adduced.

In the third place, the factors of reagent concentration, extent of surface and time of reaction are different in the two cases under consideration in a cumulative way. Bubble-machine experiments were conducted with relatively large reagent concentration, very small mineral surface and a relatively large reaction time. The effect of each of these modifications of the usual flotation procedure is to intensify many fold the action of the reagent on the mineral surface.

The authors present evidence to show that cleaved and ground surfaces yield substantially the same angle of contact upon testing in xanthate solution. More faith could be placed in the practical value of the authors' method of surface preparation if it were shown that the contact angle of cleaved and of ground surfaces is the same in distilled water. However, the authors state that with their method of preparation all the minerals tested gave a zero angle of contact in distilled water, whereas they quote Taggart, Taylor and Ince as saying that it is difficult to get (cleaved) mineral particles so clean that they show no tendency at all to attach to air bubbles. Must it be inferred that in distilled water cleaved and ground surfaces are not equivalent, but that they become so after immersion in relatively strong xanthate solution?

Messrs. Wark and Cox note that contact angles under 10° cannot be determined "with any degree of certainty" by their method. It is easy to see the reason for, and to evaluate the significance of, this fact. Let it be assumed that a bubble of 5-mm. dia. is brought in contact with a mineral surface, and forms a contact angle of 10°, the circle of contact having a diameter of 1 millimeter.

The vertical component of the gas-liquid surface tension T is $T \sin 10°$, and the total force holding the bubble to the mineral is $Tl \times (0.1) \times T \sin 10° \times \frac{1000}{g}$ or

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[11] For development of this theory, see A. M. Gaudin: Flotation, 88–98. New York 1932. McGraw-Hill Book Co.

4.0 mg. In comparison it might be noted that the buoyancy of the bubble amounts to $\frac{1}{6}\pi \times (5)^3$ or 66 mg. Clearly the vertical component of the resultant of the surface tensions is no match for the buoyancy of the bubble. Hence, lack of contact may well be observed upon inadequately gentle withdrawal of the bubble from the mineral surface. These considerations apply, of course, with even greater emphasis in the case of contact angles of the order of 1°, or less, should such contact angles exist.

Assume again that a cubical galena particle 30 microns in diameter (about 500 mesh) adheres to a bubble with a contact angle of 10°, substantially all of one face being in contact with the bubble. The apparent weight of the particle, which has to be supported by the vertical component of the resultant of the three interfacial tensions, is $(0.003)^3 \times (7.5 - 1.0) \times 1000$, or 0.00017 mg. Comparatively the force holding the bubble to the mineral is enormous, since it is

$$4 \times 0.003 \times 72.8 \times 0.174 \times {}^{1000}/_{980} = 0.155 \text{ mg.}$$

or roughly 1000 times as large.

It has been pointed out[12] that, owing to agitation in flotation machines, the supporting surface tension might well be empirically regarded as the difference between the surface tension of pure water and the actual surface tension of the pulp—rather than the surface tension of the pulp itself. This difference in surface tensions is much smaller than the surface tension of the pulp, being of the order of 1 to 5 per cent as large. Substituting 0.728 dyne for 72.8 dynes in the estimation of the force holding the bubble to the mineral, the latter becomes 0.00155. This is still some nine times as large as the weight to be supported.

It does appear, therefore, that after allowing for agitation in the flotation cell, 500-mesh galena would in all probability float under conditions which imply a contact angle so small as not to be measurable with the bubble machine. The same, however, would not be true of 65-mesh, nor even of 150-mesh galena, for which flotation would require either larger angles of contact or a greater surface-tension depression of the pulp solution as compared with water, or both.

It is a strange coincidence, if not a particularly significant fact, that depressed sulfides are substantially more floatable than gangue in the size range of 400 to 4000 mesh. That, in the absence of collector, sulfides like galena and chalcopyrite are floatable in the size range of 200 to 4000 mesh, but not at sizes coarser than about 200 mesh, is even more suggestive of the existence of a small but significant contact angle, and of the simultaneous absence or utter insignificance of such an angle at the boundary between gangue, gas, and solution.

On the assumption of a contact angle small enough to be undetected by the bubble machine, yet large enough to have a significant bearing on flotation operation, the scientific observations of Messrs. Wark and Cox can be reconciled with laboratory and operating evidence that no practicing metallurgist will deny.

The comments that have just been presented may be summarized in the form of a generalized hypothesis:

1. For a mineral to be floatable it is necessary that the solid-liquid-gas contact angle be not zero; but it is not necessary that the angle be large enough (10°) to be determinable by the present type of bubble machine.
2. Larger contact angles connote floatability at an increasingly large size.
3. In most laboratory and practical operations the contact angle of the floating mineral is small enough not to be accurately measurable by the present type of bubble machine.

Evidence in favor of the hypothesis is as follows:

1. Coarse sphalerite is not floated by ethyl xanthate, but fine sphalerite (400 to 2000 mesh) is floated, even in the presence of free cyanide ion, although fine gangue

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[12] Reference of footnote 11, 154.

is floated to a much smaller extent. In this instance, part of the sphalerite is floated mechanically between bubble walls, and part of it by adherence to gas bubbles, or else its recovery should not exceed that of gangue. This is shown both by plant data and by laboratory experiments.[13]

2. Fine galena is floated in the absence of collector, but galena is said to give no measurable contact angle in pure water.

3. Most sulfides are floated by methyl xanthate in the absence of activator, but they give no measurable contact angle in methyl xanthate solution.

4. Sphalerite (up to 100 mesh) is floated by moderately large amounts of amyl xanthate in the absence of activator, but it gives no measurable contact angle in amyl xanthate solution.

5. Considerable time is required for a contact angle to develop with galena if the concentration of xanthate is small (around one part per million). Yet xanthate at that concentration, and acting on a surface of mineral thousands of times larger, has an unmistakable immediate effect on the flotation of galena.

If a specimen prepared by grinding according to the method of Wark and Cox were immersed in a galena-gangue pulp, with the usual amount of reagents, and for the usual, very short period of conditioning, and then an attempt were made at measuring the contact angle, it is likely that the angle would be recorded as nil.

G. Barsky, Warners, N. J. (written discussion).—The findings of the authors that different gases produce no difference in contact angle is quite significant. The authors themselves have expressed surprise at their results. The view that they were not measuring actual contact of the gas-liquid interface but rather contact angle between hydrocarbon film on the mineral and a film of oriented xanthic acid in the bubble wall would explain their results. The recent work of DeWitt and Roper[14] is interesting in this connection. DeWitt and Roper point out that "at the surface of an aqueous solution of xanthate . . . Theoretically, the concentration of the surface should be greater than in the bulk of the liquid; such is shown to be the case in Table V." These authors continue: "However, we do not know whether we are measuring the interfacial concentration of the potassium salt, the acid or the ions; the most plausible assumption is that we are measuring the combined effect of all three."

Most of the measurements made by Messrs. Wark and Cox were at a pH of 6–6.5. Xanthic acid is an extremely weak acid and its alkali salts hydrolyze readily. At a pH as low as 6–6.5 there is considerable hydrolysis to the free acid. The data reported by the authors in Table 8 showing the effect of potassium xanthate concentration on the critical pH value may be used to substantiate the view that the contact angle measured was that of a xanthic acid film and not of the gas. Xanthic acid being a weak acid, we may write the equation:

$$\frac{X^- \times H^+}{HX} = K \text{ (ionization constant for xanthic acid).}$$

If we assume that in every case the same quantity of xanthic acid was required to give the contact angle, the product of the values of hydrogen ion concentration times the xanthate ion concentration should be a constant, within the limit given by the authors for the pH determinations, which is 0.2. The figures for hydrogen ion concentration and xanthate ion concentration may be taken from the authors' values, using the concentration of potassium xanthate as given, since potassium xanthate is very largely ionized. Calculation of these products shows:

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[14] *Jnl.* J. J. Brunner: Master's thesis, Montana School of Mines, June, 1931.

[13] Amer. Chem. Soc. (1932) **54**, 444.

Conc. KEtX Mg. per Liter	pH	Conc. H^+	Product Conc. $EtX^- \times H^+$
500	11.9	$10^{-11.9}$	6.5×10^{-4}
100	11.0	$10^{-11.0}$	10×10^{-4}
25	10.2	$10^{-10.2}$	16×10^{-4}
5	9.1	$10^{-9.1}$	40×10^{-4}

The agreement between the first three products is within the limits of the accuracy of the determinations of pH, the factor 1.6 corresponding to $10^{0.2}$. This would seem to substantiate the theory advanced. The discrepancy with the lowest concentration of xanthate is probably due to the oxidation of some of the xanthate. With the small quantity used, the percentage oxidized might be considerable. Moreover, we have neglected the xanthate transformed to un-ionized xanthic acid and removed as heavy metal salt. If corrections could be applied for the xanthate ion removed by oxidation and formation of xanthic acid, etc., the agreement in the products would be still better.

Alkali is not as effective in preventing critical contact with amyl xanthate as with ethyl xanthate, since amyl xanthic acid is a weaker acid and the hydrolysis of its salts takes place at higher values of pH^+.

Although in most of their experiments the authors used concentrations of xanthate approximating those employed in actual flotation, the pH of their solutions was much lower. Apparently this was necessary in order to obtain satisfactory results. On the basis of the theory mentioned above, satisfactory measurements of the contact angle should be possible at higher values of pH^+ if small amounts of pine oil are used to provide the film in the bubble wall. It is hoped that the authors will extend their work to include such measurements. It is significant that the authors could not obtain the same critical pH values in 25 mg. per liter KEtX solutions with drops of organic liquids.

In their study of the effect of alkali cyanide on the contact angle at pyrite surfaces in Table 9, the authors have overlooked the fact that, hydrogen cyanide being a very weak acid, low pH results in the formation of hydrocyanic acid, which is very slightly ionized. Consequently the effect of low pH is to decrease the concentration of cyanide ion present. This is the explanation of the fact that the critical pH was lowered when larger quantities of cyanide were used. No recourse to explanation involving cyanide iso-cyanide equilibrium or hydroxy-cyanide or carbonate-cyanide complexes is necessary. In addition to the effect of pH on the cyanide ion concentration there is, of course, another effect, that of the pH on the xanthate ion-xanthic acid equilibrium, which would, of course, have an effect, since the concentration of potassium xanthate was kept constant.

L. J. Christmann, Linden, N. J. (written discussion).—On page 227 the authors say "As far as we know, no such direct evidence has previously been advanced concerning the orientation of adsorbed films on solid surfaces. Orientation in the films of the fatty acids on water, however, has been proved and a similar orientation has been tacitly assumed for solids by several writers on flotation." The authors are not the first to bring direct evidence concerning orientation of adsorbed films on solid surfaces. Hardy has shown that in lubrication heteropolar compounds are adsorbed by metals as oriented films. He says:[15] "When normal paraffins and their related normal acids and alcohols are studied as lubricants a singular fact emerges . . . the friction of solid faces lubricated with one of these acids or alcohols takes from five minutes to an hour or more to come to a steady state.

"Immediately after the lubricant is applied, the friction is high, and throughout the 'latent period' it falls until the final steady value is reached. Now the molecules of these substances . . . are chains of carbon atoms loaded at one end with $-COOH$ or $-CH_2OH$ group. The attraction of the two ends of the molecule for the solid face is certain to differ greatly, and the latent period may be supposed to be the time occupied in reaching the maximum degree of orientation of molecules of the lubricant at the face.

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[15] J. Alexander: Colloid Chemistry—Theoretical and Applied, 300–301. New York, 1926. Chem. Catalog Co.

"If this supposition be correct, lubricants composed of *molecules whose ends are alike should show no latent period.* Such are the normal *paraffins*, and actually *surfaces lubricated with one of these do not under any circumstance exhibit the remarkable delay in reaching a steady state.* The first observed value is the same as the last."

According to Traube,[16] both Woog[17] and Trillat[18] showed that fatty acids show the same orientation on metals as they do on water; *i.e.*, the *polar group towards the metal* and the *nonpolar group towards the air.*

The work of the authors shows that the above is also true for minerals, a result which was to be expected. Indeed, the delay shown in the authors' experiments in reaching the equilibrium angles is a striking parallel to the observations of Hardy.

On page 190, the authors say, "It is obvious that the primary requisite for flotation is sticking between air and mineral, or more strictly, a sufficient attraction between mineral and air for air to be able—partly at least—to replace water at the mineral surface."

We believe that this statement is based upon a misconception of the flotation mechanism. It is *not obvious* that the primary requisite is sticking between air and mineral; what we believe to be a primary requisite for flotation is the sticking to the mineral of an oriented hydrocarbon film of promoter, and that this hydrocarbon film is then soluble in the hydrocarbon film of the froth.

When this is understood such experimental facts as those reported by the authors—that the so-called contact angle is the same for the various minerals (with but few exceptions) with a given xanthate—follows of necessity. What the authors are measuring is not the contact angle at the line of triple contact, air-mineral-water, but of air-hydrocarbon surface-water.

The authors themselves show that the mineral is covered with an oriented xanthate film. This can be represented schematically as follows:

$\boxed{\text{Mineral}}$—XR

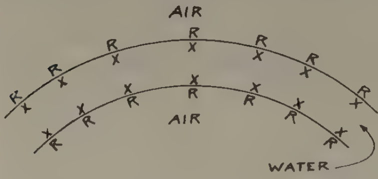

FIG. 7.—CONSTITUTION OF AIR BUBBLE.

Langmuir and Harkins have shown that at an air-water interface a heteropolar compound orients as follows:

$$\frac{\text{Air}\quad R}{\text{Water}\ X}$$

This means that an air bubble must be constituted as indicated[19] in Fig. 7.

The R of the film on the mineral is soluble in the R of the froth film, so that a mineralized froth would be constituted as in Fig. 8.

On page 204, we find a plot of contact angle versus number of carbon atoms in the alkyl group. The results do not apply to actual flotation practice. At the laboratory of the American Cyanamid Co. cetyl xanthate was found to be a very poor promoter. Messrs. Swainson and Anderson[20] pointed out that as the number of carbon atoms in the alkyl group of the xanthate increases, the promoter effect of the xanthate under actual flotation conditions increased up to five carbon atoms, above which there was a marked decrease. This has been further substantiated by Siedler,[21] who found that the promoter activity of alkyl xanthates for galena reaches a maximum when the alkyl group contains 5 to 6 carbon atoms, above which a marked drop occurs. This is to be expected, since the polarity and orientation decrease with increase in the number of carbon atoms in the alkyl group.

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[16] *Metall und Erz* (1928) **25,** 618.

[17] *Der Ölmarkt* (1927) **9,** 153.

[18] *Metallwirtschaft* (1928) **7,** 101.

[19] See American Cyanamid Co. *Tech. Paper* 17.

[20] *Trans.* Amer. Electrochem. Soc. (1931) **60,** 329.

On page 210, the authors mention that the time for development of contact angle in some cases required 2 hr. Did the authors notice any relationship between rate of development of contact angle and the number of carbon atoms in the alkyl group of the xanthates?

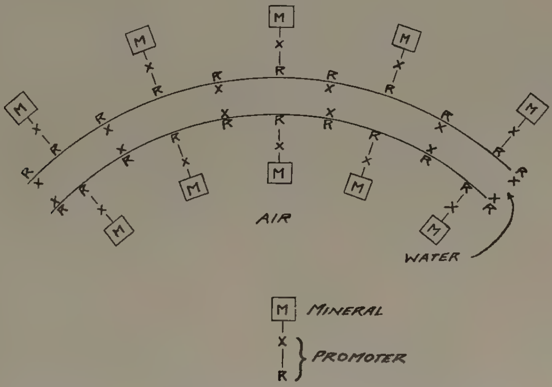

FIG. 8.—CONSTITUTION OF MINERALIZED FROTH.

On page 208, the authors say that ethyl xanthate is without apparent effect on sulfur. Sulfur is nonpolar and is not wet by water; on the other hand, if some pine oil is added to the sulfur it is readily wet by water. In this case the orientation of pine oil (if we represent it as RX) is as follows:

$$\boxed{S}-RX$$

The polar group orients toward the water and the nonpolar group towards the sulfur. It is well known that sulfur floats better when nonpolar compounds such as kerosene and benzol are used than with the usual promoters and frothers.

I. W. WARK AND A. B. COX (written discussion).—Since they deal in a constructive way with some apparent divergences between practical flotation tests and bubble-machine experiments, Professor Gaudin's comments are specially valued. Complete agreement was never expected. The main objective of our tests was a study of the surface reactions of sulfide and gangue minerals with some of the more common flotation agents and of how these reactions influence air-mineral contact. It was stated explicitly that other factors affect flotation, and in so far as there are differences between bubble tests and flotation tests, these differences indicate that a search for other contributing factors is necessary. Nevertheless evidence which is being accumulated indicates that the factor of most importance in flotation is the possibility of air-mineral contact.

The only serious divergence cited by Professor Gaudin between the bubble-machine tests and those of actual flotation tests centers about the floatability of galena and other sulfide minerals in the absence of xanthates. Such floatability is, as Professor Gaudin states, in apparent disagreement with our contention that *clean* surfaces of sulfide minerals are not air-avid. For this apparent discrepancy he offers an explanation in the form of a working hypothesis which should be useful in directing further experimental work. We offer certain alternative hypotheses which need testing before Professor Gaudin's hypothesis can be accepted.

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[21] *Metall und Erz* (1931) **28**, 425–29.

Professor Gaudin suggests correlation of the trigonometric functions of contact angle with the change in thickness of the hydrocarbon coating. The function of value in this connection is the cosine—or better, $(1 - \cos \theta)$, which is the function determining the tenacity of contact. Table 11, deleted in the condensation of the paper, demonstrates that though each higher member of the series leads to better shielding, the additional shielding due to each additional CH_2 group decreases rapidly as the length of the hydrocarbon chain increases. The suggested extension to include other series of compounds had already been undertaken and the results will be published in the near future.

TABLE 11.—*Characteristic Contact Angles*

Alkyl Group	Methyl	Ethyl	n-Propyl	n-Butyl	iso-Butyl	iso-Amyl	Cetyl
θ	50°	60°	68°	74°	78°	86°	96°
$1 - \cos \theta$	0.36	0.50	0.63	0.72	0.79	0.93	1.10

In an attempt to explain the apparent discrepancies already mentioned, Professor Gaudin questions whether the surfaces used in the bubble machine are of the same nature as those in flotation pulps. It is scarcely true, however, that we have taken it for granted that ball-mill grinding produces comminution by attrition, for it is clearly stated in our paper that both cleaved and ground surfaces need investigation. Professor Gaudin's contention is that we have not sufficiently investigated cleaved surfaces. Our reason for preferring ground instead of cleaved surfaces was that with the latter there is an uncontrolled variable, which is well illustrated in Table 1 of the paper by Taggart, Taylor and Ince; namely, that the condition of cleaved surfaces is so variable that the angle of contact varies between zero and 35°. On the other hand, ground and polished surfaces invariably give a zero angle of contact in pure water. Our Table 1, in which every surface used was first tested in distilled water and gave a zero contact angle, proves that the method of grinding makes no difference, a conclusion for which Professor Gaudin asks our evidence. Evidence has been adduced, both experimental and from the literature, to show that many cleaved surfaces are also air-repellent, and it may be added that such surfaces appear clean under the microscope. It is perhaps well to emphasize that our aim has been to obtain "clean" surfaces such as have more recently been defined by Professor Gaudin:[22] "By a clean surface is not meant a surface that is absolutely free of any alteration, but rather a surface that is as clean as can be made, and one that is reproducible." It would be fortunate if also we have succeeded in obtaining unaltered sulfide surfaces or, better still, surfaces similar to those in a flotation pulp.

Though we agree with Professor Gaudin that the smoothness and extent of the surface influences the results, we should expect (as explained on p. 210) the larger plane surfaces to coalesce with air bubbles less rapidly than the smaller angular particles of the flotation pulp. Moreover, their extent and their not being in motion place them at a distinct disadvantage with respect to speed of reaction with xanthate when compared with the small rapidly moving particles of a flotation pulp. The xanthate concentrations cited by us are not as high as the initial values used in many concentration plants. It is very doubtful, therefore, whether the conditions chosen in our work intensify the action of the xanthate to the extent suggested by Gaudin.

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[22] Reference of footnote 11, 34.

Professor Gaudin's analysis to prove that there would be little chance of detecting small angles of contact by the bubble machine neglects the highly significant fact that the buoyancy of the bubble is already completely balanced by the bubble holder. Though it was stated that angles of contact of 10° cannot be measured with certainty we did not wish to imply that they could not be detected. Contact angles as low as 10° have, in fact, been reported by Taggart, Taylor and Ince.[23] The point at issue is whether *under the same surface conditions* a small particle might attach itself to a bubble more readily than a large particle to a large bubble supported so that it has no tendency to leave the surface. In this connection a statement deleted in the condensation of the paper might have saved misunderstanding. Owing to the buoyancy effect mentioned by Professor Gaudin, it follows "that the maximum size of bubble which can be held on the mineral surface (freely suspended) is a function of the contact angle." A much smaller bubble than that taken by Professor Gaudin would be necessary for stable contact, without support of the bubble by its holder, assuming an angle of contact of 10°. We cannot agree with Professor Gaudin's statement that the diminution of, rather than the actual, surface tension is the "supporting" tension. Though this diminution is of importance with respect to frothing, the tenacity of contact is governed by the actual surface tension of the solution.

Professor Gaudin's concluding generalised hypothesis is divided into three parts. The essential parts of the first two are implicitly contained in our term for the tenacity of sticking; namely, $T(1 - \cos \theta)$. The third part, that, "In most laboratory and practical operations the contact angle of the floating mineral is small enough not to be accurately measurable by the present type of bubble machine," is the crux of his contribution to the discussion. It is here that exceedingly critical experimental work is needed. Three sources of uncertainty must be guarded against. The first two, which follow from the paper under discussion, are, firstly, that activators must be rigidly excluded and, secondly, that exposure to air in the absence of water is inadmissible. The third is that many of the common frothing agents are also mild collectors. Professor Gaudin's own work suggests that terpineol is a collector under some conditions. Recent unpublished experiments have demonstrated that pine oil and various constituents of eucalyptus oil (cineol, phellandrene and piperitone) possess mild collecting properties, though under ordinary conditions the added collector is dominant. The rates of reaction of these frothers are low under the conditions of the bubble-machine test, but the results indicate that care is necessary in drawing conclusions when such oils are present.

In order that the evidence advanced by Professor Gaudin in favor of his hypothesis should be convincing, it would be necessary in his items 1 to 3 to show:

Item 1.—That no activator unaffected by cyanide (*e. g.*, Pb^{++}) was present and that the frother had no collecting power.

Item 2.—That no collector was accidentally present, that the frother had no collecting power and that there had been no exposure of the reactive surfaces to air.

Item 3.—That no activators were present.

Further:

Item 4.—It was not claimed by us that sphalerite gives no measurable angle in amyl xanthate solution (p. 213).

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[23] Reference 1 in Bibliography.

Item 5.—Generally only a fraction of a minute is necessary for the development of contact, but longer is necessary for the establishment of equilibrium. Our paper may have been misleading here as figures were cited only for galena, which reacts most slowly and is moreover subject to a condition (p. 211) that may prevent contact despite a xanthate coating. Repeating our test of Table 6 with 1 mg. per liter of potassium xanthate, and either wiping the surface with linen or agitating the specimen with fine sand, the interfering condition did not develop and contact was possible within five minutes; shorter times have not been tried.

Professor Gaudin has suggested, finally, that the rate of adsorption of the xanthate film on one of our test specimens of galena should be determined under the conditions ruling in a flotation cell. Of the mills listed in Mayer Schranz's book on flotation, few use xanthate alone as collector, and of these the St. Joseph Lead Co., Hughesville, Mont., uses least; namely, 156 grams per ton of galena. Shaking by hand a mixture of ground quartz and galena at a pulp density of 50 per cent solids with a polished specimen of galena of about 2 c.c. volume, a blank test in water showed no tendency for galena-air contact to occur. The addition of 9 grams potassium ethyl xanthate per ton of galena led to sufficient adsorption within 10 minutes to cause the usual angle of contact (60°). There is no doubt that some contact could have been obtained earlier. Since the consumption of xanthate was less than one-fifteenth of that used in the mill referred to above, it would seem that our method of testing, when modified suitably, is capable of dealing with concentrations as low as those of practical operations and that even with lower concentration the response is rapid.

Though we cannot accept some of his views, we wish to thank Dr. Barsky for constructive and stimulating criticism of several points in our paper.

On page 210 it is shown that the xanthate ions which are concentrated at the air-water interface are orientated with the active (*not* the hydrocarbon) group towards the water. Xanthic acid and its potassium salt would be orientated similarly. It is thus difficult to see how, as suggested by Dr. Barsky, a complete orientated film of xanthic acid could effect contact with the alkyl groups of the adsorbed xanthate molecules or ions on the mineral surface. It is orientated in the wrong direction to help contact and would, in fact, hinder contact. It must be remembered, moreover, that the film is not complete. The work of De Witt and his coworkers shows that 25 mg. per liter of potassium ethyl xanthate has no significant effect on the surface tension of water. The interface must therefore consist largely of air and water molecules; *i. e.*, water and air must actually be in contact. These facts compel rejection of Dr. Barsky's hypothesis. It is presumed that Dr. Barsky does not suggest that there is a liquid film of xanthic acid surrounding the air bubble. Xanthic acid is too soluble for this to occur.

It is questionable whether xanthic acid is an extremely weak acid. A solution containing 2½ grams per liter of highly purified potassium salt is scarcely if at all hydrolyzed, the pH value being 6.9. (On long standing the pH value rises but this is due to decomposition of xanthic acid.) Pending a determination of the dissociation constant we prefer to regard it as a relatively strong acid. The figures of the fourth column of Dr. Barsky's table are incorrect; the figures should be 4×10^{-15}; 6.25×10^{-15}; 10×10^{-15} and 25×10^{-15}. Let it be assumed tentatively that the departure from constancy in these figures is due to experimental error; *i. e.*, that the apparent constancy is not fortuitous. At least three different interpretations are possible; namely:

1. Dr. Barsky's view. This is in opposition to evidence on pages 217 to 220, that all minerals do not give identical critical pH values.

2. The development of air avidity by the minerals may be due to adsorption of xanthic acid itself, and for each mineral there is a minimum or critical concentration below which none is adsorbed.

3. Since $[EtX^-] \times [H^+]$ = a constant = 10^{-14}, and since $[OH^-] \times [H^+] = 10^{-14}$ it follows that for these four solutions $[EtX^-]/[OH^-]$ = a constant = 1. This would be expected if the function of the alkali was simply to replace the xanthate at the surface according to the equation $PbX_2 + 2OH^- = 2X^- + Pb(OH)_2$. More experimental evidence is required, however, before the apparent constancy can be regarded as significant.

We are perhaps to blame for Dr. Barsky's misunderstanding in regard to the pH values of the solutions used. The value 6.5 is that of a 25 mg. per liter solution of potassium ethyl xanthate in distilled water. Satisfactory results can be obtained in solutions of higher pH value. Using chalcocite, contact is easily effected in N-NaOH solutions. In following out a program of introducing, one at a time, the more important variables of flotation, a start has already been made in measuring the effects on contact of frothers of definite composition. Cresol and amyl alcohol do not influence the magnitude of the contact angle due to potassium ethyl xanthate. This fact appears to disprove Dr. Barsky's theory concerning the part played by frothers in bringing about contact. Certain other reagents commonly described as frothers, for example, pine oil and o-toluidine, do influence contact angle, but they themselves are collectors in the absence of xanthates, and it is as collectors that they bring about contact. We have not yet determined whether frothers alter the critical pH value for galena.

Dr. Barsky's explanation of the influence of alkalis in conjunction with cyanides is substantiated by some later work which will be published shortly.

The work cited by Dr. Christmann does not appear to be "such direct evidence" of orientation as that presented by us. In his discussion of the paper, Professor Gaudin has been kind enough to say, "This is more direct proof of orientation . . . than had heretofore been adduced."

It is unfortunate that the use of such time-honored phrases as "air-mineral contact" and "sticking between air and mineral" should have led to a misunderstanding. In the application of thermodynamics and mechanics to flotation the deductions are valid irrespective of theories of contact and therefore it is best to omit reference to theory in describing the contact. It was clearly stated in the paper that contact between air and a clean unconditioned mineral surface is impossible, and that before contact can be effected the mineral becomes coated by a unimolecular orientated film. There should be no doubt, therefore, concerning our conception of the nature of air-mineral contact.

The hypothesis submitted by Dr. Christmann is insufficient to explain contact between mineral and air in the solutions used by us. No frother was present. We see three grave objections to the theory of flotation schematically represented in Fig. 8: (1) Flotation is partly a mechanical process and a mineral particle must receive some support other than that indicated in this figure; support which normally is obtained by the particle sinking into the liquid until a definite contact angle is established; (2) where contact is first established in the pulp or low in the froth the bubble has but one wall and, as pointed out in our reply to Dr. Barsky, the particular orientation of the frother molecules at this wall is opposed to Dr. Christmann's theory; (3) even when certain noncollecting frothers are present they do not influence contact angle.

In referring to the results for the higher xanthates, Dr. Christmann says (without evidence) that the polarity and orientation decrease with increase in the length of the carbon chain. The results cited in Table 3 are opposed to this view, for the contact angle gives a dual measure of polarity and orientation at the mineral surface. Traube's rule[24] proves also that there is the usual orientation of compounds containing long carbon chains at a liquid-air interface. Dr. Christmann says that our results do not apply in actual flotation tests. Surely the correct interpretation is that some factor other than adsorption and orientation is also of importance in flotation. We remind Dr. Christmann of the statement on page 190, that "Other factors influence flotation . . . "

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[24] Freundlich-Hatfield: Colloid and Capillary Chemistry, 64–68.

Referring to page 210, Dr. Christmann says that 2 hr. was sometimes required for development of contact angle. Our statement was that occasionally 2 hr. is required *before the angle becomes constant.* Generally though the equilibrium angle is reached more slowly with the higher xanthates, contact is more rapidly attained, and indeed an angle of 60° is far more rapidly attained in octyl xanthate solutions than in those of ethyl xanthate.

Principles of Flotation, II—An Experimental Study of the Influence of Cyanide, Alkalis and Copper Sulfate on the Effect of Potassium Ethyl Xanthate at Mineral Surfaces

By Ian William Wark* and Alwyn Birchmore Cox*

(New York Meeting, February, 1933)

In an earlier paper[1] measurements of contact angles due to the effect of xanthates on mineral surfaces were reported. The solutions in which these measurements were made differed widely from those of modern flotation circuits. It is intended, however, by introducing, one at a time, the other principal reagents of flotation, to approach gradually the solution composition of flotation liquors. The work now described is an attempt to determine the effect of one of the most important of these reagents—namely, sodium cyanide—on the establishment of air-mineral contact. The work has been carried out for the following companies: Broken Hill South, North Broken Hill, Zinc Corporation, Electrolytic Zinc of Australasia, Mount Lyell Mining and Railway, and Burma Corporation.

Since the effect of cyanide is inseparably connected with the alkalinity, the results cited below deal with the mutual effects of alkalis and cyanide. Alkalis alone exert a significant effect on the air-mineral contact but comparatively large amounts are required. Since the addition of a small amount of cyanide greatly decreases the degree of alkalinity necessary for the suppression of contact at surfaces of any of the minerals experimented with (except galena) and since alkalis are rarely used alone in practice, little attention has been paid to cyanide-free solutions. It was shown in the previous paper that, excluding the dixanthogen effect, whenever contact was practicable at all in an ethyl xanthate solution the angle of contact was 60°. There is, therefore, less necessity to measure equilibrium contact angles; in fact, the experiments herein described have generally been conducted with a view to determining whether contact is possible and not to measuring the actual value of the contact angle.

The separations which have generally to be effected in dealing with complex sulfide ores are: (1) galena from sphalerite, pyrite, etc.; (2) sphalerite from pyrite and/or gangue minerals; (3) copper minerals from pyrite and gangue minerals; (4) pyrite from gangue minerals; (5) chal-

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* Department of Chemistry, University of Melbourne, Melbourne, Australia.

[1] I. W. Wark and A. B. Cox: Page 189, this volume.

copyrite from sphalerite. Of the copper minerals, the sequel will show that chalcopyrite lies closest to pyrite with regard to the influence of cyanide and alkalis on air-mineral contact. If conditions are selected such that chalcopyrite differs from pyrite it seems probable that the differentiation between pyrite and other copper minerals (bornite, covellite and chalcocite) will be greater. It is apparent, therefore, that the minerals whose relations to cyanide are most worth investigation are galena, sphalerite, pyrite and chalcopyrite.

An activator is essential for the flotation of sphalerite by potassium ethyl xanthate. Though several activators are available, copper sulfate is invariably used in practice, and it has therefore been used in this investigation. In view of its almost universal presence in zinc-iron separations, it is necessary also to know its effect on pyrite. Moreover, as some copper invariably dissolves in the pulp liquors in which copper ores are floated, it is equally important to know the effect of copper sulfate on the chalcopyrite-pyrite separation.

The comments of Coghill and Clemmer[2] suggest that the object of work of this kind is not universally appreciated. It has two main objects. First, it is directed toward explaining the nature of the physical and chemical forces upon which the process is based, since an intelligent understanding of the process would lead to its more efficient application. That the flotation cell is unsuitable as a medium for investigating the underlying principles of flotation has been recognized by Brighton, Burgener and Gross[3] who say, "Conditions in a flotation cell are too complex to permit direct study of the reactions" (occurring therein). Secondly, more immediate practical gains might precede a complete theoretical explanation.

Because of the bewildering number of possible combinations of the variables of flotation, investigation must be restricted to those which the experimenter, with few data to go on, considers most important. The writers have studied only the variables that it was expected would most influence the contact between air and mineral, but do not thereby imply that others are unimportant. Comparison of the results cited later with practical flotation operations has, however, led to the belief that the air-mineral contact is at least as important as any other single factor in flotation. The term "air-mineral contact" must be read in its broadest significance; air-mineral contact is generally impossible in the absence of an adsorbed collector film at the mineral surface, but the term is best used without regard to theories of the mechanism of the adhesion.

REVIEW OF LITERATURE

Though there is an extensive literature dealing with the influence of the cyanide addition in practice, little is known concerning its functions, except as a deactivator for sphalerite. Its use as a depressant for pyrite is not understood. Also, it is not yet decided whether unactivated sphalerite is influenced by cyanide. Ince[4] claims that sphalerite removes cyanide from solution and Berl and Schmitt[5] and Christmann[6] suggest a mechanism for this abstraction. Gaudin, Haynes and Haas[7] claim that cyanide is not a depressant for sphalerite when used with collectors that do not require an activator, and think that no cyanide coating is formed on the surface of the sphalerite. The subject is further discussed by Wark.[8] No later attempts have been made to account for the very interesting observation of Tucker, Gates and Head that chalcocite may be depressed by cyanide in alkaline solutions.[9] The publications of these authors and of Gaudin and his collaborators[10] are the most important contributions to our knowledge of the effect of cyanide on the floatability of pure minerals in practical flotation. More recently, Brighton, Burgener and Gross[11] have concluded that one function of the cyanide is to dissolve the xanthate film from the surface, but they have postulated several other possible functions of the cyanide. Kraeber[12] has made a valuable study of the influence of cyanide on the floatability of zinc and iron minerals.

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[2] W. H. Coghill and J. B. Clemmer: Page 449, this volume.

[3] T. B. Brighton, G. Burgener and J. Gross: *Eng. & Min. Jnl.* (1932) **133**, 276.

Various modifications of a theory of preferential coating by slime or precipitated salts have been advanced by Gates and Jacobsen;[13] Tucker, Gates and Head;[14] Taggart, Taylor and Ince;[15] and Gaudin.[16] Ince is now of the opinion that the theory of slime coatings can not completely account for the action of cyanides. The influence of cyanide on dispersion is discussed by Fahrenwald[17] and by Wark.[18]

The writers of three recent textbooks on flotation[19] have had perforce to dismiss the consideration of the nature of the depression due to cyanide with short references to some of these theories. Gaudin, however, speaks with greater certainty concerning the function of cyanide as a deactivator for blende.[20]

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[4] C. R. Ince: *Trans.* A.I.M.E. (1930) **87,** Milling Methods, 261.

[5] E. Berl and B. Schmitt: *Koll. Ztsch.* (1930) **52,** 333.

[6] Christmann: Amer. Cyanamid Co. *Tech. Paper* 17 (1930).

[7] A. M. Gaudin, C. B. Haynes and E. C. Haas: Flotation Fundamentals, Pt. 4, Univ. of Utah *Tech. Paper* 7 (1930).

[8] I. W. Wark: Amer. Electrochem. Soc. *Preprint* 61-10 (1932).

[9] E. L. Tucker, J. F. Gates and R. E. Head: *Trans.* A.I.M.E. (1926) **73,** 372.

[10] A. M. Gaudin et al.: Flotation Fundamentals, pts. 1–5, Univ. of Utah (1928–30).

[11] Reference of footnote 3.

[12] L. Kraeber: *Mitt.* Kaiser-Wilhelm Inst. für Eisenforsch. (1930) **12,** 343.

[13] J. F. Gates and L. K. Jacobsen: *Eng. & Min. Jnl.-Pr.* (1925) **119,** 771.

[14] Reference of footnote 9.

[15] A. F. Taggart, T. C. Taylor and C. R. Ince: *Trans.* A.I.M.E. (1930) **87,** 285.

[16] A. M. Gaudin: *Eng. & Min. Jnl.* (1927) **124,** 1045.

[17] A. W. Fahrenwald: *Trans.* Electrochem. Soc. (1931) **60,** 311.

[18] Reference of footnote 8.

[19] A. M. Gaudin: Flotation. New York, 1932. McGraw-Hill Book Co.
E. W. Mayer and H. Schranz: Flotation. Leipzig, 1931. Herzel.
W. Luyken and E. Bierbrauer: Die Flotation. Berlin, 1931. Springer.

EXPERIMENTAL METHODS

Contact Measurements

The method of preparation of the mineral specimen and the manner of bringing the bubble into contact with the surface were described in the paper beginning on page 189. In order to obtain a measure of the effect of cyanide and alkali on air-mineral contact in the presence of a given concentration of xanthate, tests were made to determine for a series of different pH values the minimum concentration of sodium cyanide required to prevent contact. The usual procedure was to prepare xanthate solutions of a constant pH value but with increasing concentrations of cyanide and to test whether the prepared mineral surfaces when immersed in them were able to make contact with an air bubble. In this manner, two concentrations of cyanide, differing only slightly, were obtained such that at one concentration air-mineral contact could be induced and at the other (higher) concentration it could not. This procedure was repeated for a series of pH values. At the lower pH values, because of the shape of the cyanide concentration-pH curves (q.v.), it was expedient to keep the sodium cyanide concentration constant and to determine critical pH values for each sodium cyanide concentration.

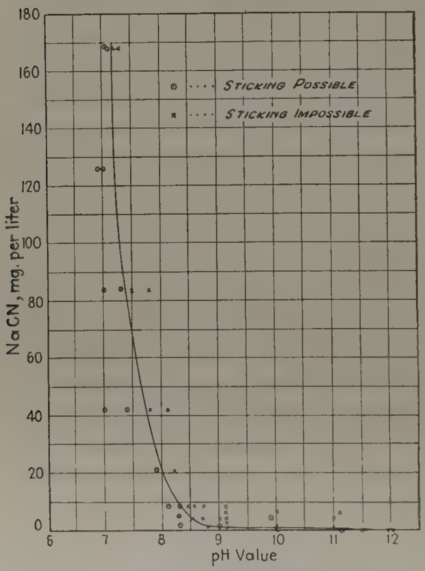

FIG. 1.—CONTACT CURVE FOR CHALCOPYRITE WITH KEtX = 25 MG. PER LITER, ILLUSTRATING THE METHOD OF CONSTRUCTION OF THE GRAPHS.

O indicates that contact with air bubble is possible; X that contact with air is not possible.

The experimental results are best recorded graphically. The method of construction of the graphs is illustrated by Fig. 1, in which the experimental points for chalcopyrite in the presence of 25 mg. per liter potassium ethyl xanthate are plotted. The pH value is plotted as abscissa and the sodium cyanide concentration as ordinate. The curve is drawn so that at points above it contact between air and mineral is not possible, whereas at points below it contact is possible. The experimental points have not been included in the other figures because where more than one curve is included they would be confusing.

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[20] A. M. Gaudin: Reference of footnote 19, 77.

Slight differences in procedure are necessary according to whether an activator is or is not necessary with the particular mineral under investigation. Where no activator was required the mineral specimen was tested 30 min. after it was placed in the solution. This time is quite arbitrary, but experience has shown that if there is no contact after 30 min. none will develop within several hours.

Where an activator was necessary, two solutions of identical pH value were prepared, the first containing all the reagents except xanthate, the second containing xanthate as well. The mineral was conditioned for 30 min. in the xanthate-free solution and then for an equal time in the xanthate-bearing solution. It was then tested in the customary manner. This procedure was preferred to adding the xanthate directly to the first solution because such addition would alter the concentrations and pH value. Moreover, this procedure conforms as closely as possible to flotation practice, where, generally, an endeavor is made to allow interaction between sphalerite and copper sulfate prior to the xanthate addition. Though the curves have been determined in this manner, subsequent tests with pyrite have demonstrated that the position would not have been altered substantially had one solution only been employed. Conditioning in a xanthate-free solution does not therefore, in this case at least, greatly influence the equilibrium cyanide effect, but it may influence the speed with which equilibrium is attained.

Precipitates form in many of the solutions. These settle on the surface of the mineral, preventing the development of the maximum angle of contact with the test bubble, though, underneath, the surface may have responded fully to the xanthate. After testing the surface in the customary manner, it has therefore been the practice to wipe the surface free from precipitate with a pad of linen and immediately to test it again before the precipitate has had time to settle. That the violent agitation in a flotation machine has an effect similar to that of wiping the surface with linen has already been demonstrated.[21]

Even when there is no visible contamination, the surface has been wiped with linen before the final test with the bubble. This procedure had perforce to be adopted in order to obtain reproducible results.

It is easy to establish contact between air and mineral while the pH value and cyanide concentration are still well below the amounts that prevent contact. Contact is established with less ease, however, when either the pH value or the cyanide concentration is only slightly less than the value necessary to prevent contact, and the angle of contact may then fall short of 60°. Apparent contact for ½ min. may be necessary before adhesion occurs between air and mineral. Longer contact between the solution and the mineral is necessary for the development of the maximum angle, but after 30 min. some contact is obtained. In this region also wiping the surface with linen becomes necessary. The region over which contact is possible but for which the full angle is not attained is relatively small.

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[21] Page 242, this volume.

Owing to the difficulty of obtaining large quantities of pure minerals, each mineral specimen has been used for a number of tests. With some of the copper minerals and with pyrite, difficulties arose out of this procedure, especially at high pH values and cyanide concentrations. Covellite in particular, and bornite and chalcopyrite to a lesser extent, are altered by contact with highly alkaline cyanide solutions. Polishing the surface is not then sufficient to restore them to their original state. The method adopted to restore the surface to the normal condition is to warm the specimen for a few minutes in normal hydrochloric acid, then to wash thoroughly in running water, and finally to grind lightly before polishing. After having determined the curves for these minerals, it has been customary to make a few check tests with new specimens.

Reagents of analytical purity were used throughout. The strength of the standard sodium cyanide solution was checked at frequent intervals. Sodium carbonate has been preferred to other alkalis for general use because of its buffering action in the pH range 8 to 10. Adjustment to the required pH was made with dilute hydrochloric acid. Usually 150 to 250 mg. per liter of Na_2CO_3 was used; this amount, judged by the considerations on page 209, is of the order generally added in practice. For pH values above 10.6 where some stronger alkali is necessary, sodium hydroxide has been used. The xanthate was purified at frequent intervals by recrystallisation from alcohol. Solutions more than 36 hr. old were discarded. Siedler[22] has shown that no appreciable decomposition occurs within this time.

The Hellige Comparator was used for the routine determination of pH values, two indicators with overlapping ranges being used whenever practicable. Certain doubtful readings were checked against the hydrogen electrode. The pH values of the less alkaline solutions change during the conditioning period. It has been customary to follow these changes by measurements during and after the tests, which were repeated if the change was more than 0.2 units. Where constancy within this limit could not be obtained, the lowest value recorded was accepted if sticking occurred and the highest value if sticking did not occur. The variation in pH value during one hour seldom exceeded 0.4 units, and by conducting several tests at slightly different initial pH values it has usually been possible to determine the position of the curve within 0.2 unit.

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[22] P. Siedler: *Metall und Erz* (1931) **28**, 425.

Since the order of mixing the reagents may be of importance (q.v.), the following uniform procedure was adopted. The solutions were prepared in a 100 c.c. flask, the order of addition of the reagents being: (1) sodium carbonate and/or caustic soda, (2) sodium cyanide, (3) hydrochloric acid to adjust the solution approximately to the required pH value, (4) distilled water to 60 c.c., (5) when necessary, copper sulfate, (6) when necessary, potassium xanthate, (7) water to 100 c.c., (8) a small fraction of a cubic centimeter of hydrochloric acid or sodium carbonate solution for the final adjustment of pH value.

Copper-ion Concentrations

Using data from Findlay,[23] it may be shown that the difference in potential, E, between two copper electrodes of which one is immersed in a 0.1 N $CuSO_4$ solution and the other in a solution in which the ionic concentration of copper is C is given by the expression below, from which, if E be measured, C can be evaluated:

$$E = 0.029 \log \frac{C}{3.7 \times 10^{-2}} \qquad [1]$$

It has been found expedient to substitute for the copper electrode in 0.1 N $CuSO_4$ a saturated calomel electrode. Allowing for the difference in potential between these two electrodes (0.045 volt) the expression for the observed potential difference becomes

$$\begin{aligned} E &= 0.045 + 0.029 \log \frac{C}{3.7 \times 10^{-2}} \qquad [2] \\ &= 0.087 + 0.029 \log C \end{aligned}$$

Because of the formation of complexes in alkaline cyanide solutions, the concentration of the copper ion is not equal to that of the added copper sulfate and expression 2 enables the ionic concentration to be determined.

In measuring E a modification[24] of the standard method has been followed, the solutions being freed from oxygen by bubbling nitrogen through the solution. That oxygen corrodes a copper electrode has been shown by Walker, Sorrels and Breckenridge;[25] it is therefore advisable to remove it from the solution. Kreuger and Kahlenberg[26] have demonstrated that with the inert gases nitrogen, hydrogen, argon and helium a common potential is obtained at a copper electrode whereas, particularly in alkaline solutions, the presence of oxygen gives an abnormal result; this again suggests that oxygen should be removed. Steady potential differences were obtained within 30 min. of the introduction of the electrode, nitrogen being bubbled continuously. With pH values below 9, nitrogen sweeps out some carbon dioxide and the use of sodium carbonate as a buffer had to be discontinued.

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[23] A. Findlay: Practical Physical Chemistry, 209. London, 1925.

[24] I. W. Wark: *Jnl.* Chem. Soc. (1923) **123**, 1824.

[25] W. M. Walker, J. H. Sorrels and J. M. Breckenridge: *Trans.* Amer. Electrochem. Soc. (1925) **48**, 47.

[26] A. C. Kreuger and L. Kahlenberg: *Trans.* Amer. Electrochem. Soc. (1930) **58**, 107.

Waxes were dispensed with in mounting the copper electrode because they influence the results in strongly alkaline solutions, which decompose them. The electrodes were mounted in such a manner that the solution had access to the electrode at the liquid-gas line; in the absence of oxygen, this is not harmful.

Since xanthate is adsorbed by copper, the electrode could not be used in its presence and the results cited later are all for xanthate-free solutions. Measurements were made at room temperature. In the more alkaline solutions they were reproducible within one centivolt and in the less alkaline solutions within a few millivolts. The attainment of greater accuracy would have involved a long preliminary research.

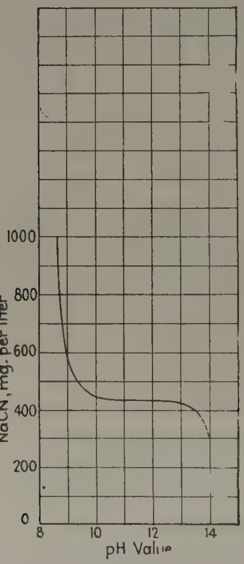

Fig. 2.—Contact curve for chalcocite. KEtX = 25 mg. per liter.

Experimental Results

Influence of Alkalis and Cyanide for Various Minerals.—The results for various minerals are plotted in Figs. 1 to 3. Difficulty has been experienced in fixing the positions of the lower left-hand portions of the pyrite and chalcopyrite curves. The pyrite curve in this region may be 0.5 pH units in error, but with chalcopyrite the error of the determination is smaller. With the other minerals, and with these two elsewhere along their curves, it is possible to duplicate results more accurately. Evidently some uncontrolled factors influence the results for pyrite and chalcopyrite in the regions of uncertainty. These factors may be the ionic concentration of iron or copper. Both may be expected to vary according to the rate of solution of the mineral and any impurities therein. The order of these fortuitous variations is not sufficiently high to cause doubt as to whether any of the curves cross; there is a well marked differentiation between any two of them.

Influence of Added Copper Sulfate.—The results for various minerals are plotted in Figs. 4 to 6.

Effect of Order of Addition of Reagents.—The condition of a flotation liquor may depend upon the order in which the constituents are added and upon the time interval between the additions. Thus, in a solution containing 84 mg. per liter NaCN, 300 mg. per liter Na_2CO_3, 150 mg. per liter $CuSO_4.5H_2O$ and 25 mg. per liter potassium ethyl xanthate (KEtX), whose pH value is 10.7, it was found that no precipitate was formed if the cyanide and xanthate were mixed before the copper sulfate addition. If, however, other orders of mixing were adopted, it was possible to produce

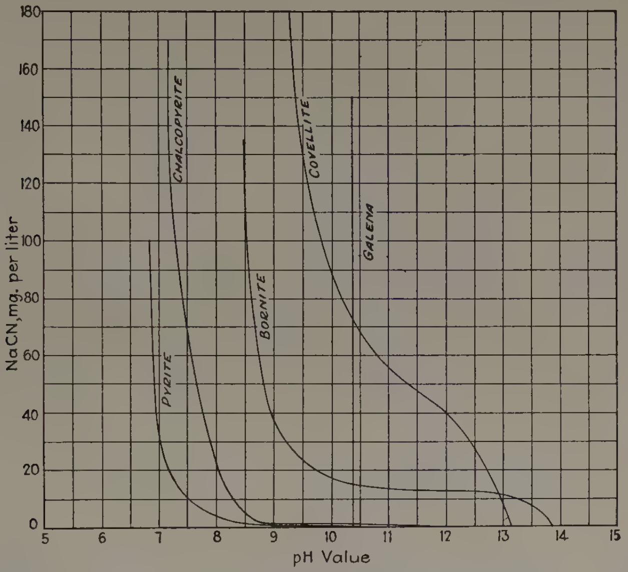

FIG. 3.—CONTACT CURVES FOR PYRITE, CHALCOPYRITE, BORNITE, COVELLITE AND GALENA. KEtX = 25 MG. PER LITER. CONTACT POSSIBLE ONLY BELOW THE CURVES.

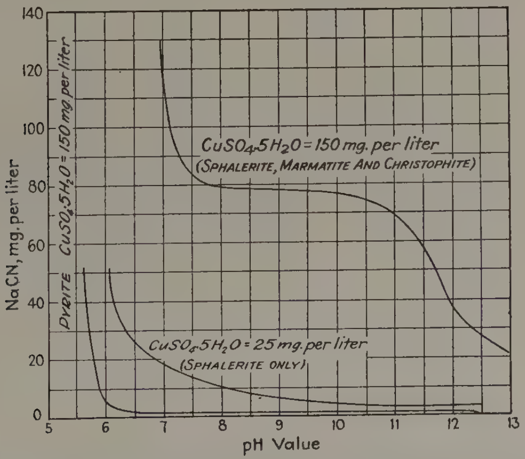

FIG. 4.—CONTACT CURVES FOR SPHALERITE, MARMATITE, CHRISTOPHITE AND PYRITE IN THE PRESENCE OF COPPER SULFATE. KEtX = 25 MG. PER LITER.

either a bluish white precipitate or a deep yellow precipitate and both were stable enough to exist for several days. Sometimes, however, the bluish white form dissolved or changed to the yellow. On thermodynamical grounds it follows that ultimately the same equilibrium

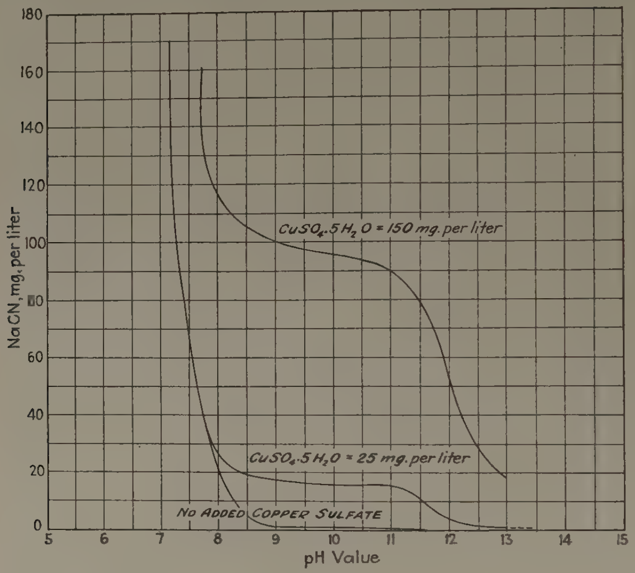

FIG. 5.—CONTACT CURVES FOR CHALCOPYRITE WITH VARIOUS ADDITIONS OF COPPER SULFATE. KEtX = 25 MG. PER LITER.

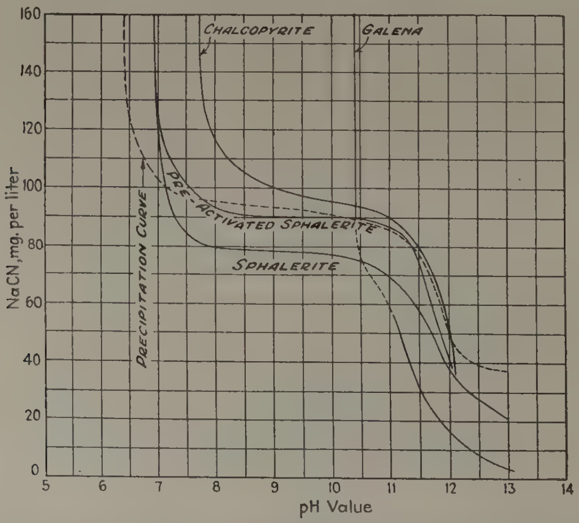

FIG. 6.—PRECIPITATION CURVE FOR COPPER XANTHATE; CONTACT CURVES FOR SPHALERITE, CHALCOPYRITE AND GALENA; AND CONTACT CURVE FOR SPHALERITE PREACTIVATED BY COPPER SULFATE. $CuSO_4.5H_2O$ = 150 MG. PER LITER AND KEtX = 25 MG. PER LITER FOR ALL CURVES.

condition must be attained irrespective of the order of addition of reagents. Some of these solutions are therefore in a metastable condition. In solutions in which the xanthate and cyanide were first mixed, chalcopyrite sometimes failed to respond to an air bubble under conditions in which it would have responded had the xanthate been added after the copper sulfate, but the displacement of the curve was small. The influence on flotation is therefore probably relatively small.

Effect of Nature of Alkali Added.—In adopting the above method of plotting results, it has been assumed that the alkali influences contact only through its influence on the hydroxyl or hydrogen-ion concentrations. It is implied, therefore that alkalis other than sodium carbonate would be equally effective at the same pH value. This has been found to be true in a few cases where the addition of other alkalis has been tested. Experimental verification has been confined to a few tests with bornite and chalcopyrite and the tests already reported (p. 189) for galena in the absence of cyanide. The analysis of the results for copper minerals and pyrite in the next section is further justification for the choice of pH value as a variable in plotting the results.

Influence of Frothers on the Cyanide and Alkali Effects.—Dr. G. Barsky has suggested[27] that the frother plays a more important part in flotation than has been assigned to it in that paper. It is suggested by Dr. Barsky that there is a coherent film of orientated frother between the xanthate-conditioned mineral and the air bubble and that such a film is essential in flotation. Should a film of this type assist in the establishment of air-mineral contact, it would necessarily influence the contact angle. Further, Dr. Barsky suggests that the presence of a frother would influence the critical pH value for galena. There is a possibility also that a film of the type suggested would modify the influence of cyanide on the air-mineral contact due to xanthates. The following series of tests, with frothers present, give no evidence of the presence of such a film.

The frother does not influence the magnitude of the contact angle in the following cases:

1. Galena and ethyl xanthate with 30 mg. per liter pine oil, 75 mg. per liter iso-amyl alcohol, 100 mg. per liter o-cresol, or 50 mg. per liter terpineol.
2. Galena or chalcopyrite and potassium di-ethyl dithiocarbamate with 100 mg. per liter o-cresol.
3. Galena and potassium amyl xanthate with 100 mg. per liter o-cresol.
4. Chalcopyrite and ethyl xanthate with 50 mg. per liter terpineol, 100 mg. per liter o-cresol, or 75 mg. per liter iso-amyl alcohol.

It has been found also that the critical pH value for galena in the presence of 25 mg. per liter potassium ethyl xanthate is not appreciably altered by the presence of 50 mg. per liter o-cresol or of 50 mg. per liter iso-amyl alcohol. Moreover, the presence of 50 mg. per liter o-cresol does not influence the position of certain points on the cyanide-pH curves for bornite and activated sphalerite. Iso-amyl alcohol and terpineol were also without influence. It is probable, therefore, that noncollecting frothers are without significant effect on the positions of the curves given in this paper.

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[27] Page 236, this volume.

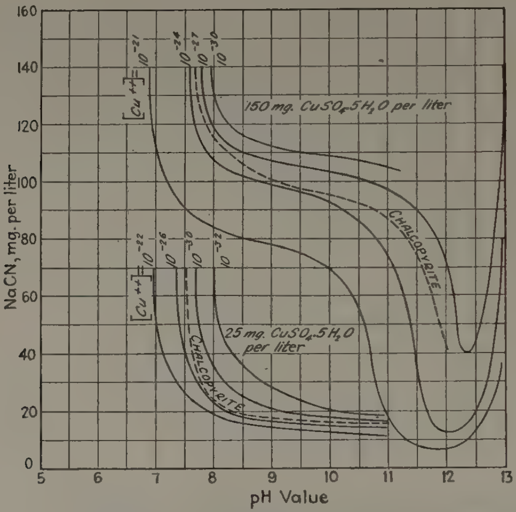

FIG. 7.—COMPARISON OF CONTACT CURVES FOR CHALCOPYRITE WITH CURVES OF CONSTANT COPPER-ION CONCENTRATION.
Continuous lines, copper-ion concentration; broken lines, chalcopyrite-air contact.

Apparently the function of frothers in flotation is the purely physical one of providing a large air-water interface.

Certain frothers are also collectors. Thus o-toluidine leads to contact between air and a chalcopyrite surface in the absence of xanthate or other recognised collector. Such collector-frothers, if added in addition to xanthates, might be expected to modify the positions of the cyanide-pH curves.

Copper-ion Concentrations.—The results are plotted in Fig. 7, in which a series of lines of equal copper-ion concentration show the manner in which additions of cyanide and alkali affect the copper-ion concentration. The upper series of lines are for solutions containing 150 mg. per liter $CuSO_4.5H_2O$ and the lower series for solutions containing 25 mg. per liter. There are included for comparison the contact curves for chalcopyrite in the presence of the same amounts of copper sulfate.

DISCUSSION OF RESULTS

It follows from the figures that in so far as selectivity depends upon air-mineral contact a complete separation of the common sulfide minerals is theoretically possible. Careful control of the xanthate, cyanide and alkali concentrations and careful exclusion of other variables would be essential. In practice, however, it would not be feasible rigidly to exclude other variables. Zinc, iron and copper, etc., find their way into the circuit liquors by oxidation and/or solution of the minerals in the ore; the frother used often has collecting properties itself; there may be intimate association between the minerals. These and other factors tend to prevent perfect separations and before the maximum practical efficiency of separation could be estimated their influence would have to be determined. Nevertheless, except in so far as association prevents it, there is no obvious reason why complete separations should not be aimed at.

Considering first the copper-free solutions, galena alone of the minerals tested is uninfluenced by the addition of cyanide. Irrespective of the amount of cyanide, up to 1 gram per liter at least, the critical pH value is 10.4 ± 0.2. It was suggested in the preceding paper that this behavior of galena might be connected with the singular inability of lead to form complex cyanides.

In considering the results for the copper minerals and pyrite, use has been made of a fact to which attention was directed by Dr. Barsky; namely, that hydrocyanic acid being a weak acid, the cyanide-ion concentration is greatly influenced by the presence of alkalis. He further suggested that only the portion of the added sodium cyanide that is present as cyanide ions is of importance with regard to inhibition of contact in xanthate solutions. Accepting Madsen's value for the dissociation constant of hydrocyanic acid,

$$\frac{[H^+]\,[CN^-]}{[HCN]} = 4.7 \times 10^{-10} \text{ at } 18°\text{C}.$$

these views satisfactorily account for the influence of the alkali on the amount of cyanide required to prevent contact between an air bubble and the copper and iron minerals. Thus the pH value when using sodium cyanide does not appear to be a direct factor in determining contact, but rather an indirect one, in that it influences the actual cyanide-ion concentration. Using the above equation, the percentage of the added sodium cyanide that is present as cyanide ions can be calculated for each pH value. Table 1 shows the actual cyanide-ion concentration per 100 mg. per liter of added sodium cyanide. Using this table, the actual

TABLE 1.—*Influence of pH Value on Cyanide-ion Concentration*

pH Value	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0	11.0	12.0	13.0
Milligrams CN^- per 100 mg. NaCN added..............	0.0249	0.0788	0.248	0.777	2.38	6.87	17.0	31.7	43.8	52.0	53.0	53.1

cyanide-ion concentrations of solutions of compositions represented by points on the curves of Figs. 2 and 3 have been calculated. In Table 2 the first and second columns for each mineral give the pH values and NaCN concentrations at several points along the respective curves; the figures of the third columns are the corresponding calculated cyanide-ion concentrations. The contact curves thus prove to be lines of substantially constant cyanide-ion concentration.

For bornite the degree of constancy is good, considering that there was a 30,000-fold variation in hydrogen-ion concentration. The degree of constancy with the other minerals is not as good. This may, of course, be due to an inadequacy of the explanation but it is by no means certain that the variation is not due to experimental error. No great order of accuracy is to be expected in the more alkaline regions for chalcopyrite and pyrite. Here, since the steps in the experimental variations in cyanide concentration were 1 mg. per liter, there is a possible error of 0.5 mg. per liter in the position of the curve, thus introducing a possible error of from 30 to 50 per cent in the calculated cyanide-ion concentration. An insufficient number of solutions was tested to determine the curve for chalcocite with great accuracy. More points were determined for the covellite curve and the fact that this crosses the bornite curve suggests that, for this mineral at least, there is a second contributory factor. Experiments of greater refinement are necessary to determine whether the apparent departures from complete agreement with the above theoretical interpretation are due to experimental error.

However, it appears from Table 2 that for each mineral there is a critical cyanide-ion concentration above which contact is impossible and below which contact is possible. Approximate values are, for a 25 mg. per liter KEtX solution:

	CN^-, Mg. per Liter		CN^-, Mg. per Liter
Chalcocite	180	Chalcopyrite	0.4
Covellite	34	Pyrite	0.1
Bornite	7		

The significance of these facts is not clear. Tentatively, it might be assumed that cyanide ions are themselves adsorbed when the concentration exceeds these figures. But even this explanation is incomplete, for it cannot be stated whether cyanide displaces xanthate according to a definite chemical equilibrium, whether a simple or a complex cyanide is adsorbed, or why the critical cyanide-ion concentration should differ from mineral to mineral. It is, however, not essential to assume adsorption of the cyanide. An alternative assumption may be advanced which satisfactorily "explains" the facts; namely, that the cyanide dissolves from the surface of the minerals those atoms which normally bind the xanthate to the surface. This is almost equivalent to saying that the cyanide dissolves copper or iron xanthate from the surface.

TABLE 2.—*Influence of Cyanide-ion Concentration on Contact*

Chalcocite			Covellite			Bornite			Chalcopyrite			Pyrite		
pH	NaCN Added, Mg. per Liter	CN^-, Mg. per Liter Calc.	pH	NaCN, Mg. per Liter	CN^-, Mg. per Liter Calc.	pH	NaCN, Mg. per Liter	CN^-, Mg. per Liter Calc.	pH	NaCN, Mg. per Liter	CN^-, Mg. per Liter Calc.	pH	NaCN, Mg. per Liter	CN^-, Mg. per Liter Calc.
8.7	1000	101	9.0	235	40.0	8.5	100	6.9	7.2	150	0.49	6.8	100	0.157
9.0	600	102	9.5	130	41.2	9.0	37	6.3	7.5	67	0.52	7.0	31	0.077
9.5	480	152	10.0	88	38.6	10.0	17	7.4	8.0	21	0.50	7.5	10	0.078
10.0	450	197	11.0	55	28.6	11.0	13	6.8	8.5	5.0	0.34	8.0	3.5	0.083
11.0	440	229	12.0	40	21.2	12.0	13	6.9	9.0	2.0	0.34	8.5	(1.5)	(0.103)
12.0	435	230	13.0	(7)	(3.7)	13.0	12	6.4	10.0	(1)	(0.44)	9.0	(<1)	(<.17)
13.0	430	228							11.0	(<1)	(<0.52)	10.0	(<1)	(<.4)
Mean		177			33.9			6.8			0.44			0.10

The extreme right-hand ends of the curves have been neglected in this consideration. Here all the curves dip rather suddenly to meet the horizontal axis at a definite pH value. This has been defined (*Tech. Pub.* 461) as the "critical pH value" of the mineral for the particular xanthate and concentration used. At lower pH values cyanide is necessary to prevent contact; at higher pH values contact is impossible even in the absence of cyanide. The critical pH value for chalcocite has not been determined.

Except for the crossing of the covellite and bornite curves, the critical pH values are in the same order as the critical cyanide-ion concentrations. The former may be read from the graphs.

Copper sulfate was present in the tests represented in the remaining figures, and because of the presence of complex copper cyanides it is not possible to calculate the concentration of the cyanide ion by the above method. Making certain assumptions, however, with regard to the composition of the complex, an attempt has been made to explain the action of the cyanide in certain cases.

Fig. 4 shows that, despite their differing iron content, sphalerite, marmatite and christophite are equally sensitive to the influence of alkaline cyanide solutions. On the other hand, the curve for pyrrhotite is not identical with that for these minerals.[28] The christophite used, which was obtained from Germany by courtesy of Herr Ing. Miksch, contained 13.1 per cent iron, of which only 0.75 per cent was present as pyrite, and the ratio Zn:Fe (excluding pyrite) was 2.8:1.

The present work is in agreement with the view that the function of the cyanide in preventing the flotation of sphalerite is to remove the copper-bearing film from the surface if it has been activated previously, or to prevent its activation if copper is present. Based on contact measurements, certain evidence has been obtained which supports the de-activation view. This is summarized in Table 3.

It has been suggested[29] that when activated by copper sulfate, sphalerite becomes coated with a film of covellite. It was therefore of interest to compare the curves obtained for sphalerite specimens known to be activated by copper sulfate with the curve for natural covellite.

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[28] On page 200 it was stated that pyrrhotite does not respond to ethyl xanthate, but that it is activated by copper sulfate. Recent tests have shown, however, that the critical pH value for pyrrhotite in a 25 mg. per liter KEtX solution is close to 6.0. There was no response to a KEtX solution of this strength in distilled water because the pH value was slightly higher—approximately 6.5. The addition of copper sulfate, owing to its hydrolysis, lowered the pH value sufficiently to enable the pyrrhotite to respond. Whether the copper sulfate has any other function has not yet been determined, but it certainly does not activate pyrrhotite in the manner that it activates sphalerite.

[29] Reference of footnote 7.

The sphalerite was preactivated by immersion for 10 min. in 150 mg. per liter $CuSO_4.5H_2O$ solution; no more effective activation was obtained by immersion for 12 hr. in a 500 mg. per liter solution. In determining the curves both for the covellite and the preactivated sphalerite, the pH value and cyanide content of the solutions were varied in the usual manner and all solutions contained 150 mg. per liter $CuSO_4.5H_2O$ and 25 mg. per liter KEtX. The curve for preactivated sphalerite is given in Fig. 6. The curve for covellite lies very much higher; it was not determined accurately. These results do not prove that sphalerite is not coated by a thin film of covellite, but they do prove that whatever the nature of the film, it has not the properties of a surface of massive covellite.

TABLE 3.—*Cyanide as a Deactivator of Sphalerite*

Test No.	Consecutive Immersion of Sphalerite in Different Solutions (Specimens Water-washed before Each Immersion)			Contact Angle
1	20 mg. per liter $CuSO_4.5H_2O$	25 mg. per liter KEtX		60°
2	25 mg. per liter KEtX	20 mg. per liter $CuSO_4.5H_2O$		Nil
3	20 mg. per liter $CuSO_4.5H_2O$	150 mg. per liter NaCN	25 mg. per liter KEtX	Nil
4	150 mg. per liter NaCN	20 mg. per liter $CuSO_4.5H_2O$	25 mg. per liter KEtX	60°

The curve determined thus for preactivated sphalerite lies above that of Fig. 4, which was obtained by allowing preactivation for 30 min. in a solution containing the same amount of copper sulfate but containing also the amounts of cyanide and alkali specified by the position on the curve of the point under consideration.

Fig. 4 shows that with only 25 mg. per liter copper sulfate added, the quantity of cyanide necessary to prevent contact between sphalerite and an air bubble is very much less than when 150 mg. per liter has been added. Preactivation in a solution of copper sulfate in water also raises the curve for sphalerite in the presence of 25 mg. per liter $CuSO_4.5H_2O$. The horizontal portion of the curve (between pH values of 8 and 10.5) then corresponds to approximately 16 mg. per liter.

The influence of the concentration of copper sulfate on the position of the curve for chalcopyrite is shown in Fig. 5. Though the left-hand portions of the two lower curves are shown as coinciding in this figure, more refined experiments would probably reveal some slight differentiation. Copper sulfate diminishes the potency of the added cyanide. With pyrite, however, for pH values below 8, the influence of copper sulfate is in the other direction. (See Figs. 3 and 4.) For pH values above 8, on the other hand, 150 mg. per liter of copper sulfate raises the curve for pyrite slightly, and 25 mg. per liter does so even more, though by no means to the same extent as it raises the chalcopyrite curve. Over the whole range of pH values, therefore, it seems likely that the presence of copper sulfate is helpful with regard to the pyrite-chalcopyrite separation. Whether the optimum amount is normally present in flotation liquors, and whether the separation at any given plant would benefit by direct addition of copper sulfate, would depend upon the other reagents added and upon the conditions ruling there.

Gaudin and Taggart and their respective collaborators, and the writers also, have endeavored to trace for a given solution a relationship between the adsorption of a xanthate film at the surface of a mineral and the formation of a precipitate of a heavy metal xanthate in that solution. The curve (Fig. 6) showing the relationship between the amounts of cyanide and alkali necessary to prevent the precipitation of copper xanthate has therefore been determined for constant copper sulfate and xanthate additions of 150 and 25 mg. per liter respectively. At points above the curve there is sufficient cyanide to prevent precipitation; at points below the curve precipitation occurs. Sometimes precipitation is delayed and there is often difficulty in distinguishing between a yellow xanthate, a yellow cyanide, two bluish white precipitates, probably containing both xanthate and cyanide, and a green mixture of some of these. The latter usually changes slowly to one of the other forms. The precipitation curve was determined for solutions that had been left for 30 min. after mixing; probably equilibrium was not reached in many cases. A complete phase-rule study is necessary to determine the position of this curve and the nature of the precipitates.

In Fig. 6 the horizontal portion of the curve for fully activated sphalerite almost coincides with the corresponding section of the precipitation curve. It is probably more than a coincidence that here the ratio $NaCN:CuSO_4$ is very close to but slightly greater than 3:1. (The sodium cyanide concentration corresponding to this ratio is 88 mg. per liter.) It is suggested that the significance of this fact is that a complex cuprocyanide ion is formed and tentatively one might adopt the formula $Cu(CN)_3^-$ for this ion, which would be formed according to the equations

$$2NaCN + CuSO_4 = Na_2SO_4 + Cu(CN)_2$$
$$Cu(CN)_2 + NaCN = NaCu(CN)_3 = Na^+ + Cu(CN)_3^-$$

It is possible that the complex splits up into cyanogen and a cuprocyanide $NaCu(CN)_2$, or in some other way, but this is immaterial in the present discussion, which postulates only a $NaCN:CuSO_4$ ratio of 3:1.

It is only when a slight excess over the amount of cyanide necessary to convert all the copper to this complex ion has been added that the copper-ion concentration is reduced sufficiently to prevent the formation of a precipitate, and seemingly, also, this excess of cyanide can dissolve the surface film responsible for the activation of the sphalerite.

This view is supported by the fact that if only one-sixth of this amount of copper sulfate be added, that is, 25 mg. per liter, the curve for preactivated sphalerite indicates that one-sixth the amount of cyanide (approximately 16 mg. per liter) suffices to cause the removal of the film. Here again the precipitation curve for copper xanthate lies close to the curve for preactivated sphalerite.

The supposed formation of the ion $Cu(CN)_3^-$ also enables a quantitative explanation to be given for the displacement of the chalcopyrite curves by the addition of copper sulfate. Fig. 1 demonstrates that for each pH value a specific concentration of sodium cyanide is required to prevent contact between mineral and air. If 25 mg. per liter $CuSO_4.5H_2O$ is added, Fig. 4 shows that there is required an approximately constant increase in sodium cyanide concentration of 15 mg. per liter, which is independent of the pH value over a comparatively wide range. This is the concentration theoretically necessary to convert the added copper sulfate to the complex ion cited above. The added copper sulfate cannot therefore be said to activate the chalcopyrite; it merely combines with the cyanide and prevents it from depressing the chalcopyrite. A similar explanation is apparently also applicable for additions of 150 mg. per liter $CuSO_4.5H_2O$, but the quantitative agreement is not as good in this case.

If this explanation is adopted, not only must the horizontal portion of the chalcopyrite curves be lines of constant cyanide-ion concentration, but each must also be a line of constant cupricyanide-ion concentration. It then follows from the equation of the formation of the complex, namely $Cu^{++} + 3CN^- = Cu(CN)_3^-$, that the copper-ion concentration along the curves must also be constant. Fig. 7 shows that this deduction is approximately true. The copper-ion concentrations have not been determined with great accuracy; consequently this evidence should be regarded as only semiquantitative support for the above theory.

This explanation ceases to be valid for the ends of the curves, where they are no longer approximately horizontal. In the more alkaline regions ($pH > 11$) some of the copper is precipitated as hydroxide or basic carbonate and less cyanide need be added to prevent air-mineral contact. In the more acid regions also, the explanation apparently fails, but the reason is not apparent.

It might have been anticipated that the curves for sphalerite would also prove to be lines of constant copper-ion concentration, but a comparison of Figs. 4 and 7 indicates that they are not. The deviation of the sphalerite curve from the expected position may be accounted for qualitatively by considering the zinc-ion concentration, which is of equal importance with the copper-ion concentration in determining the course of the reaction leading to activation; *viz.* $Cu^{++} + ZnS = Zn^{++} + CuS$. Because of the formation of zincates in strongly alkaline solutions, fewer zinc ions are available to oppose activation than in neutral solutions; activation is therefore possible with lower copper-ion concentrations in the more alkaline solutions. It might also have been anticipated that the determined precipitation curve for copper xanthate would be a line of constant copper-ion concentration. That it is not may be due to supersaturation in the less alkaline solutions and to the precipitation of compounds other than cupric xanthate.

In Fig. 6 there is also included a curve for galena, obtained, like the other curves of this figure, in the presence of 150 mg. per liter $CuSO_4.5H_2O$. Comparison with Fig. 3 shows that only at pH values above 10.4 has copper sulfate any effect on contact. Above 10.4 the effect is similar to but slightly less than that produced by copper sulfate on sphalerite. It is deduced that galena also is coated by a copper-bearing film. At pH values below 10.4 the formation of this film is presumably prevented by the concentrations of cyanide which suffice to prevent activation at a sphalerite surface, but prevention of film formation on the galena leaves the original surface exposed to the action of xanthate and Fig. 3 indicates that it will then respond, whatever the cyanide concentration. This curve is of value in confirming the above view of the influence of cyanide on sphalerite, for apparently galena and sphalerite are similarly influenced by copper sulfate at pH values higher than 10.4 and it is therefore probable that they are similarly influenced below 10.4. If this is so, the galena curve proves that there is no copper film on the surface of either the galena or the sphalerite when the cyanide concentration is above 90 mg. per liter. This seems to exclude the possibility that the prevention of contact in the case of sphalerite is due to a cyanide-ion concentration high enough to depress the copper-coated sphalerite in a manner qualitatively similar to that by which cyanide depresses chalcopyrite.

There remains to be considered the influence of additions of copper sulfate on the contact at a pyrite surface. Comparison of Figs. 3 and 4 reveals the fact that copper itself is not a depressant for pyrite, for the critical pH value of pyrite in a 25 mg. per liter KEtX solution (cyanide being absent) is raised from 10.5 to between 12 and 13 by the addition of 150 mg. per liter $CuSO_4.5H_2O$. It also appears that along the left-hand portion of the curve of Fig. 4 there is an insufficient cyanide-ion concentration to prevent contact by the same process that prevents contact in the absence of copper sulfate. Therefore, since the prevention of contact can be due to neither the copper sulfate nor the sodium cyanide, it seems probable that together they are effective because of the cupricyanide ions which are produced from them. If this is the explanation, the pyrite curve of Fig. 4 is a line of constant cupricyanide-ion concentration. A decrease in the amount of added copper sulfate would tend to decrease the cupricyanide-ion concentration; this would raise the position of the curve in the manner already mentioned. The effect of variation in the amount of copper sulfate added, between the limits 25 mg. per liter and 1 gm. per liter, is, however, comparatively small.

The tentative theories presented in this paper may prove incapable of explaining future data. They have been presented because they may be useful in determining the course of future work. The theoretical study followed the experimental; this is the reason why certain obvious deductions from the theoretical section have not yet been tested.

SUMMARY

1. The joint influence of alkalinity and cyanide concentration on the contact between air and certain sulfide minerals in 25 mg. per liter potassium ethyl xanthate solution has been investigated.

2. Galena is unaffected by cyanide. The amount of cyanide required to depress the copper and iron minerals at any given pH value increases in the following order: pyrite, chalcopyrite, bornite, covellite, chalcocite.

3. It seems probable that the influence of the added cyanide is, for the copper and iron minerals, dependent only upon the cyanide-ion concentration. Critical cyanide-ion concentrations just sufficient to prevent response to 25 mg. per liter potassium ethyl xanthate are given for these minerals.

4. There is a critical pH value for each mineral, above which, even in the absence of cyanide, the response to 25 mg. per liter of potassium ethyl xanthate is prevented. Below this critical value the pH value appears to be important only in so far as it controls the cyanide-ion concentration.

5. In the presence of copper sulfate as activating agent, sphalerite, marmatite and christophite are equally sensitive to cyanide and/or alkali. Pyrrhotite is not activated by copper sulfate in the same manner as these minerals.

6. The higher the copper sulfate concentration, the greater is the capacity of sphalerite and chalcopyrite to withstand the action of cyanide. It is suggested that copper sulfate and sodium cyanide mutually decrease the copper-ion and cyanide-ion concentrations by the formation of the complex cupricyanide ion, $Cu(CN)_3^-$.

7. Galena is activated by copper sulfate in solutions in which the pH value is higher than the critical value.

8. Above pH = 8.5 pyrite is activated by 150 mg. per liter $CuSO_4.5H_2O$ but in less alkaline solutions the copper apparently enhances the effect of cyanide in preventing the response of pyrite to ethyl xanthate. This is probably due to adsorption of cupricyanide ions.

9. The curve showing the influence of cyanide and alkali on the precipitation of copper xanthate lies between the curves showing their influence on air-mineral contact at surfaces of sphalerite and chalcopyrite. The contact curve for covellite indicates that it is far less sensitive than is activated sphalerite to the influence of cyanide.

10. Activation of sphalerite by copper sulfate in a neutral cyanide-free solution is more effective than in an alkaline solution containing cyanide.

11. It has been demonstrated that the contact curves for chalcopyrite in the presence of added copper sulfate are lines of substantially constant copper-ion concentration.

12. Certain frothers do not have a significant influence on the cyanide and alkali effects, nor do they influence contact angles.

Acknowledgments

The authors desire to express their thanks to the managements and staffs of the companies already enumerated for their support and encouragement. They are especially indebted to Mr. H. Hey, under whose general direction the work has been carried out, and to Prof. E. J. Hartung for providing laboratory accommodation in the Chemistry Department of the University of Melbourne.

Principles of Flotation, III—An Experimental Study of Influence of Cyanide, Alkalis and Copper Sulfate on Effect of Sulfur-bearing Collectors at Mineral Surfaces

By Ian William Wark* and Alwyn Birchmore Cox*

An attempt has been made to compare the influences of the two most widely used depressants—alkalis and sodium cyanide—and the most widely used activator—copper sulfate—on the air-mineral contact induced by five sulfur-bearing compounds: (1) sodium di-ethyl dithiophosphate (sodium Aerofloat); (2) potassium ethyl xanthate; (3) sodium di-ethyl dithiocarbamate; (4) potassium iso-amyl xanthate; (5) potassium di-n-amyl dithiocarbamate. Three different organic types are included in this group and for two of these types a higher and a lower homologue have been tested.

The study of potassium ethyl xanthate, the preliminary results of which have been described,[1] was continued until there had been obtained a comprehensive account of the influence of variations in pH value and in the concentrations of xanthate, sodium cyanide and copper sulfate on the adsorption of this xanthate by galena, sphalerite, pyrite and chalcopyrite. With concentrations of copper sulfate and collector that the work with ethyl xanthate indicated would give the most illuminating results, the other four collectors were tested in a less exhaustive way.

The same general principles determine the influence of alkalis, cyanide and copper sulfate, no matter to which type the collector belongs, but there is a gradation in the quantitative measure of such influence. In the list above, the five collectors are cited in the order of their capacity to withstand the depressants, sodium Aerofloat being the least resistant, potassium di-n-amyl dithiocarbamate the most resistant. This order coincides substantially with that of the solubilities of the heavy metal salts formed by double decomposition with the collectors, those formed from sodium Aerofloat being most soluble. There is little difference between the third and fourth members of the series, and under certain conditions their positions are reversed.

In general, then, of the three types of collectors investigated, the dithiophosphate type is most easily influenced by depressants, and the

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[1] I. W. Wark and A. B. Cox: Pages 189, 245, this volume.

dithiocarbamate type is most resistant, the xanthate type being intermediate. Within each type the power to resist the influence of depressants is increased by using the higher homologues of the series, so that the higher homologues of one type may overlap, as in the series above, with the lower homologues of a generally more resistant type.

The curves obtained for these five collectors suggest that similar results would be given by such compounds as the mercaptans, monothiocarbonates and trithiocarbonates. The trithiocarbonates and the aryl mercaptans such as thiocresol form sparingly soluble salts with the heavy metals; consequently one would expect the minerals to respond to them in much the same manner as they respond to the dithiocarbamates containing the same nonpolar group. The alkyl mercaptans and the monothiocarbonates form salts which are more soluble than those of the xanthate containing the same nonpolar group; the minerals would therefore respond to these reagents in much the same manner as they respond to the corresponding dithiophosphates. A few simple tests should determine the position of any specific collector of these types in relation to the series of five collectors already studied. It is probable also that no similar soluble sulfur-bearing collector would possess advantages of a type not possessed by one of the five collectors tested, though better quantitative differentiation might be obtained with some of them.

The four minerals, galena, sphalerite, chalcopyrite and pyrite, have been investigated. From the data obtained by varying the concentrations of a given collector and of the modifiers under consideration, it is possible to specify the conditions under which the differentiation between any two of these minerals with respect to contact with air is greatest. It has become apparent that if the concentrations of cyanide and copper sulfate were carefully chosen, a satisfactory differentiation between any two of the four sulfide minerals could be obtained with any of these collectors.

EXPERIMENTAL METHODS

The experimental work has been based on the principle that if a mineral floats in any given solution, contact must be possible, in that solution, between the mineral and an air bubble. The development of contact between air and mineral indicates the presence of an adsorbed film of the collector molecules at the surface of the mineral. The apparatus employed and the methods of conducting the experiments and of drawing the contact curves have been described.[2] Usually the transition from sticking to nonsticking occurs over a narrow range of solution compositions. Sometimes, however, the transition range broadens, and it is not possible to draw a narrow line separating the regions of full contact angle and zero-contact angle in the following cases: upper curve of Fig. 8; curves *a* and *e* of Fig. 5; and the curves for pyrite and chalcopyrite of Fig. 17. In such cases the contact curve has been drawn in the position where the contact angle is changing most rapidly. (This curve can be drawn more accurately than that separating regions of slight sticking and nonsticking, but it has the disadvantage that flotation may be possible up to 0.5 pH units to the right of the curve.)

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[2] I W. Wark and A. B. Cox: Pages 189 and 245, this volume.

The manner of presentation of the results needs further description. For each collector the influence of several variables has been considered, namely: (1) concentration of collector, (2) concentration of sodium cyanide, (3) concentration of copper sulfate, and (4) pH value.

It is impossible to express in diagrammatic form the relationship that must exist between these four variables in order that contact may be possible between air and the surface of any given mineral. Actually, however, a constant concentration of each collector has been used for most of the tests with that collector, and the relationships between the other three variables could be presented in a solid model, but the data, except in the case of ethyl xanthate, are not sufficiently extensive. For the other collectors, it is possible to present at most three planes cut from this solid model. First there is the boundary plane of the figure for which the concentration of copper sulfate is zero, and second, there is the plane for which $CuSO_4.5H_2O = 150$ mg. per liter, and in some cases the plane for which $CuSO_4.5H_2O = 25$ mg. per liter is also given.

Unless otherwise specified, stoichiometrically equivalent concentrations of the collectors were used as follows:

	Mg. per Liter
Sodium di-ethyl dithiophosphate	32.5
Potassium ethyl xanthate	25
Sodium di-ethyl dithiocarbamate	26.7
Potassium iso-amyl xanthate	31.6
Potassium di-n-amyl dithiocarbamate	42.3

The order of addition of the reagents cited on page 251 (this volume) has been retained.

The results for each collector are presented in a separate section, in which the most widely practised separations are considered in the light of the experimental results, and the differentiation between minerals is compared with the corresponding differentiation when using ethyl xanthate. Any special advantages the collector may possess are considered.

The conclusions are valid only for air-mineral contact, but experience now indicates that selective flotation is based primarily upon differences in the capacity of the several minerals (suitably conditioned) to effect contact with air. Certainly the optimum conditions for differentiation determined from experiments on air-mineral contact for ethyl xanthate correspond closely with the optimum conditions for flotation as found by mill experience.

EXPERIMENTAL RESULTS

Contact Angles

In the paper beginning on page 189, it was demonstrated that when contact is possible the angle of contact at the line of triple contact between air, mineral and water is dependent upon the particular xanthate, but not on the particular mineral used. Wark and Wark[3] have shown that for several families of sulfur-bearing collectors the angle of contact is dependent only upon the particular nonpolar group of the collector employed. Two new dithiocarbamates have since been prepared, and the contact angles for these compounds with pyrite and sphalerite provide further evidence in support of this conclusion. The angles with galena and chalcopyrite are higher, however; this may be due to the presence of the corresponding iso-compounds in small amounts. The contact angles of Table 1 were obtained with the standard concentrations of the reagents; with lower concentrations of the butyl compound, galena and chalcopyrite gave contact angles of 74° also. In general the angles of contact measured during the course of this work are in agreement with those previously cited.

Table 1.—*Angles of Contact for Dithiocarbamates*

	Angle of Contact	
	Pot. Di-n-butyl Dithiocarbamate	Pot. Di-n-amyl Dithiocarbamate
Galena	81	91
Sphalerite	73	80
Chalcopyrite	80	90
Pyrite	74	80

Potassium Ethyl Xanthate

Results for solutions containing 25 mg. per liter potassium ethyl xanthate have been presented (p. 245). In the tests to be reported other concentrations of xanthate were used: (1) in copper-free solutions, (2) in solutions containing various concentrations of copper sulfate. A few other tests with the standard xanthate concentration (25 mg. per liter) have been included in the report on copper-free solutions.

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[3] I. W. Wark and Wark: *Jnl. Phys. Chem.* (1933) **37**, 805.

Influence of Alkalis

An increase in the concentration of the xanthate decreases the capacity of alkalis to prevent contact at mineral surfaces. Table 2 shows the influence of xanthate concentration on the critical pH value necessary to prevent contact at the mineral surface.

TABLE 2.—*Influence of Xanthate Concentration on Critical pH Value*

KEtX, Mg. per Liter	Critical pH Value			
	Galena	Pyrite	Chalcopyrite	Sphalerite Previously Activated
5	9.1	10.1	11.5	12.7
25	10.4	10.5	11.8	13.3
100	11.0	10.9	12.3	
625	12.5	11.8	>13.0	

Influence of Cyanide

Copper-free Solutions.—Figs. 1 and 2 show the influence of an increase in the xanthate concentration on the position of the contact curves for chalcopyrite and pyrite respectively. For both minerals the influence of xanthate concentration is surprisingly small except where the curves turn sharply upwards as the neutral point is approached; the influence of xanthate concentration is greater, however, for pyrite than for chalcopyrite.

It has been suggested that some of the sphalerite floats with the galena in practice because soluble copper salts derived from the ore tend to activate it before it reaches the flotation division. Cyanide added during grinding or ahead of the lead flotation either prevents activation or interferes with the effect thereof. The lower curve of Fig. 3 shows for preactivated sphalerite the relationship between the amount of sodium cyanide and the pH value necessary to prevent contact with air. Preactivation was effected by standing the specimen for 10 min. in a 150 mg. per liter $CuSO_4.5H_2O$ solution (a lower concentration would have been equally effective). The specimen was washed in running water before it was placed in the xanthate-bearing test solution. No copper was added to the latter solution. A very low concentration of cyanide in the test solution suffices to prevent contact with the sphalerite.

The data presented on page 258, were insufficient to indicate the nature of the process by which cyanide prevents contact at mineral surfaces. It now appears that the cyanide prevents adsorption of the xanthate film without otherwise changing the surface, except for sphalerite previously activated by copper sulfate. This has been deduced from experiments in which a curve for chalcopyrite similar to the 25 mg. per liter KEtX curve of Fig. 1 was roughly determined, using a drop of benzene instead of a bubble of air. The curve coincided—at least

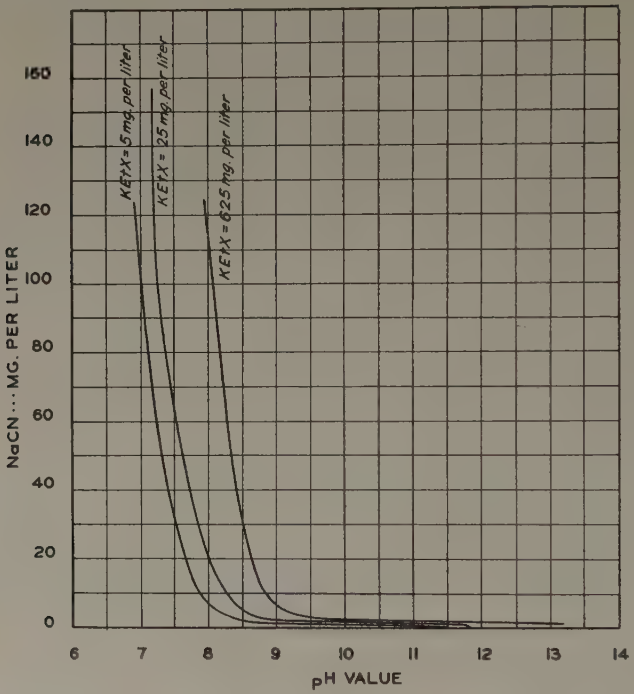

FIG. 1.—INFLUENCE OF CONCENTRATION OF ETHYL XANTHATE ON CONTACT CURVES FOR CHALCOPYRITE.

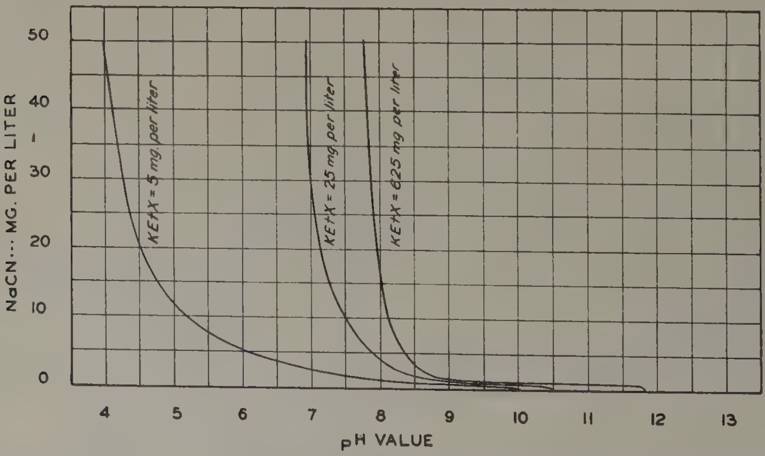

FIG. 2.—INFLUENCE OF CONCENTRATION OF ETHYL XANTHATE ON CONTACT CURVES FOR PYRITE.

approximately—with the corresponding air-mineral contact curve, but the angles of contact were to the left of the curve 115 ± 5° and to the right 75 ± 5°. The latter value is identical with that obtained for chalcopyrite and benzene in the absence of cyanide and xanthate. However, addition of a considerable excess of cyanide produced a visible precipitate on the surface, and even when this was removed by wiping the surface with linen, some invisible change prevented contact between the chalcopyrite and benzene. Similar relationships (and identical angles) were obtained for pyrite, except that even a considerable excess of cyanide did not prevent the pyrite-benzene contact.

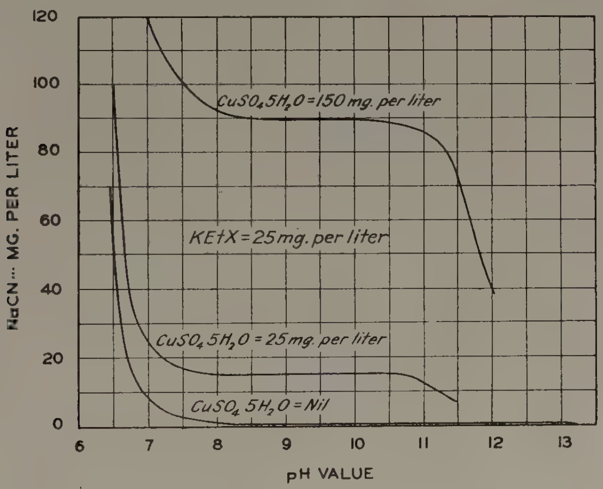

FIG. 3.—INFLUENCE OF CONCENTRATION OF COPPER SULFATE ON CONTACT CURVES FOR SPHALERITE PREVIOUSLY ACTIVATED BY COPPER SULFATE.

The prevention, by cyanide, of the adsorption of xanthate is probably connected, as suggested in the preceding paper, with its ability to form complex ions with copper, zinc and iron; the metallic ion concentration in equilibrium with the complexes is exceedingly low and may well be so low as to prevent adsorption of the xanthate (p. 212).

Copper-bearing Solutions:

(1) *Constant Xanthate Addition.*—Curves showing the influence of copper sulfate concentration for a fixed addition of 25 mg. per liter KEtX were given for sphalerite and chalcopyrite on pages 253 and 254. Corresponding curves are now given for pyrite in Fig. 4. For the reasons cited on page 264, it is considered probable that the cupricyanide ion is the active depressant for pyrite. The fact that one gram per liter of copper sulfate has but little greater influence than 25 grams per liter supports this assumption, for in both cases it is primarily the concentration of the little cyanide present, and not that of

the copper sulfate, that determines the concentration of the cupricyanide ion.

(2) *Constant Copper Addition.*—The curves considered in the preceding paragraph define the reaction of the minerals to a varying copper sulfate addition and a constant xanthate addition. The curves now to be considered define the reaction of the minerals to a varying xanthate addition and a fixed copper sulfate addition of 150 mg. per liter.

a. SPHALERITE.—Three sets of conditions have been employed to investigate the nature of the activation of sphalerite by copper sulfate: (1) The sphalerite was activated and its subsequent response to xanthate in the presence of copper sulfate and cyanide was determined; (2) the sphalerite was treated as it probably would be in the plant, that is, the copper sulfate was employed in the presence of cyanide and the xanthate was added subsequently;[4] (3) the sphalerite was treated simultaneously with cyanide, xanthate and copper sulfate at the required pH value.

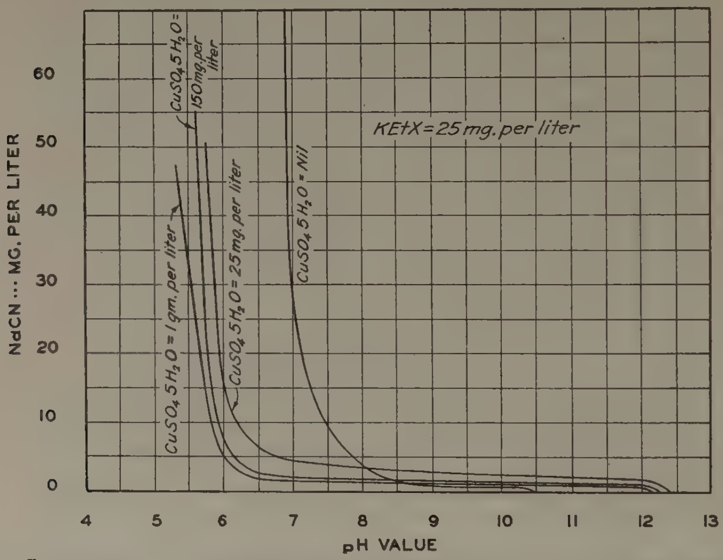

FIG. 4.—INFLUENCE OF CONCENTRATION OF COPPER SULFATE ON CONTACT CURVES FOR PYRITE.

(1) In the presence of xanthate, sphalerite already activated by copper sulfate requires an amount of cyanide dependent upon the concen-

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[4] For ease in manipulation the specimen was first treated with a solution containing both cyanide and copper sulfate at the required pH value. After a conditioning period of 30 min. the test specimen was transferred to a second solution identical in pH value and in cyanide and copper sulfate concentrations and containing xanthate in addition. Direct addition of xanthate to the first solution would have altered reagent concentrations and pH value.

tration of copper sulfate present, in order to prevent contact. Fig. 3, the top curve of which is taken from Fig. 6 on page 254, shows the required cyanide concentration for a concentration of 25 mg. per liter KEtX. The effect of variations in xanthate concentration has not been fully investigated, but curve 1 of Fig. 6 proves that at one pH value, 9.8, increasing the xanthate concentration beyond 5 mg. per liter does not alter the concentration of cyanide necessary to prevent contact.

(2) If cyanide is present with the copper sulfate it is more effective in preventing the activation of sphalerite than in destroying the activation if added subsequently, as in treatment 1. Fig. 5 shows the influence of the xanthate concentration on the amount of cyanide necessary to prevent contact. From this figure a curve may be constructed for any given pH value, showing the relationship between the concentration of the added xanthate and the concentration of cyanide that can be tolerated without preventing contact. This has been done for only one pH value, namely 9.8, and the curve is shown in Fig. 6, curve 3.

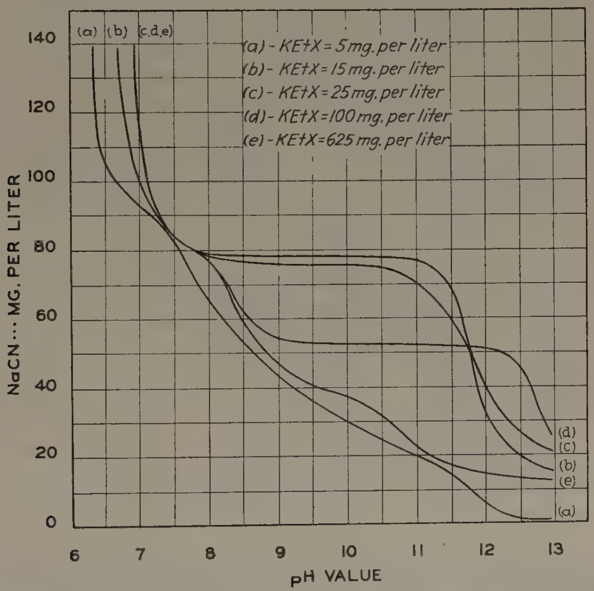

FIG. 5.—INFLUENCE OF CONCENTRATION OF ETHYL XANTHATE, IN PRESENCE OF 150 MG. PER LITER $CuSO_4.5H_2O$, ON CONTACT CURVES FOR SPHALERITE.

It is not easy to account for the decrease in the amount of cyanide required to prevent activation as the concentration of KEtX is increased beyond 15 mg. per liter (Figs. 5 and 6). That it is a question of non-activation has been proved by subsidiary tests, and is suggested, moreover, by the fact that the curves for preactivated sphalerite are higher than those for sphalerite not preactivated. It is suggested that, because of the relatively insoluble nature of copper xanthate, high xanthate concentrations (in conjunction with cyanide additions) may, by the mass action principle, reduce the copper-ion concentration to such a low value that activation does not occur.

(3) With a xanthate concentration of 25 mg. per liter, the curve for condition 3 coincides almost exactly with that for condition 2. Only at one pH value, 9.8, has the influence of xanthate concentration been determined, and if it does not exceed 50 mg. per liter curve 2 of Fig. 6 is identical with that for condition 2. There is thus, for xanthate concentrations within the range of practical application, no significant difference between conditions 2 and 3. Since the technique is simpler for condition 3 this has been selected when dealing with the other collectors, but for them there may be greater differences between conditions 2 and 3.

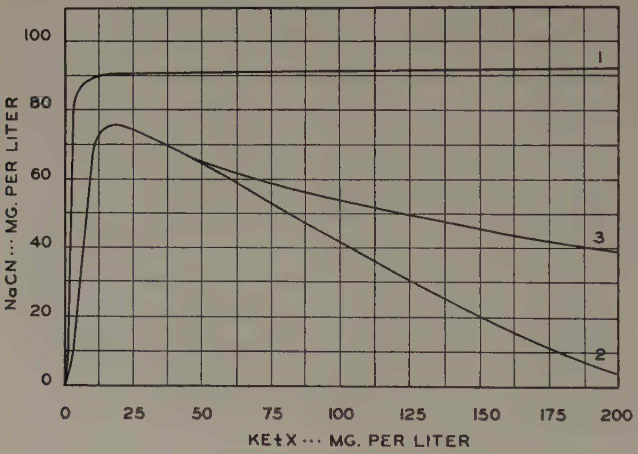

FIG. 6.—INFLUENCE OF CONCENTRATIONS OF ETHYL XANTHATE AND SODIUM CYANIDE, IN THE PRESENCE OF 150 MG. PER LITER $CuSO_4.5H_2O$ AND AT pH = 9.8, ON CONTACT AT SPHALERITE SURFACES.

1. Previously activated by copper sulfate.
2. Not previously activated.
3. Previously treated in 150 mg. per liter $CuSO_4.5H_2O$ solutions with cyanide concentrations and pH values as specified.

Figs. 5 and 6 indicate that with 150 mg. per liter $CuSO_4.5H_2O$ present, the optimum xanthate addition for the development of contact between sphalerite and an air bubble is close to 15 mg. per liter. This concentration is close to that found most effective at many flotation plants. The standard concentration previously adopted (25 mg. per liter) gives results for sphalerite almost identical with those for the optimum concentration (15 mg. per liter). This concentration (25 mg. per liter) or its stoichiometrical equivalent has been retained therefore as standard in future tests with this and other collectors. Curve 2 of Fig. 6 demonstrates that it is better to err on the high side of the optimum concentration than on the low.

b. CHALCOPYRITE.—Only one set of conditions, that corresponding to condition 3 for sphalerite, has been investigated. One would anticipate little or no difference between this case and the other sets of conditions 1 and 2 investigated for sphalerite. Fig. 7 shows that as the xanthate concentration is raised there is a progressive slight increase in the amount of cyanide and/or alkali necessary to prevent contact. In the pH range 8 to 11,[5] within which most flotation plants operate, the influence of the concentration of the xanthate is small, unless concentrations greatly in excess of those used in practice are taken.

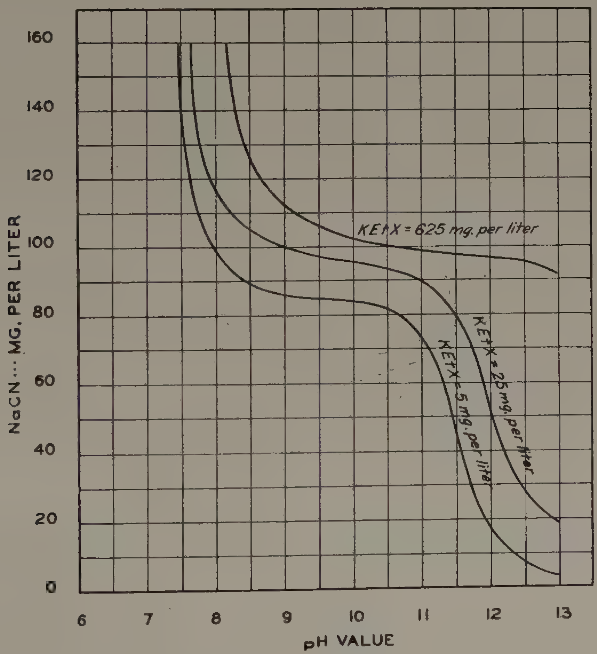

FIG. 7.—INFLUENCE OF CONCENTRATION OF ETHYL XANTHATE IN PRESENCE OF 150 MG. PER LITER $CuSO_4.5H_2O$ ON CONTACT CURVES FOR CHALCOPYRITE.

c. PYRITE.—For pyrite, also, only one set of conditions, again condition 3, has been investigated. The results, given in Fig. 8, indicate that the factors that primarily determine the positions of the curves for pyrite are not the same as those that determine the curves for chalcopyrite. (The curve for 625 mg. per liter KEtX has been represented by a broken line because experimental difficulties prevented the determination of its position as accurately as the other curves.) Comparison with Fig. 4 shows that in the presence of 25 mg. per liter KEtX, 150 mg. per liter $CuSO_4.5H_2O$ decreases the amount of sodium cyanide required to depress pyrite, whereas with either 5 or 625 mg. per liter KEtX, the same amount of copper sulfate decreases the efficacy of sodium cyanide as a depressant. In no case, however, is the effectiveness of the cyanide decreased to such an extent that the differentiation between pyrite and sphalerite, or pyrite and chalcopyrite is threatened.

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[5] In the pH range 8 to 11 the influence of pH value on the amount of cyanide that can be tolerated is generally much smaller than at other pH values. It is indeed practically negligible. Fortuitous variations are therefore less important, and this is true whatever the mineral used. It is probably partly for this reason that most flotation operations are carried out within this range.

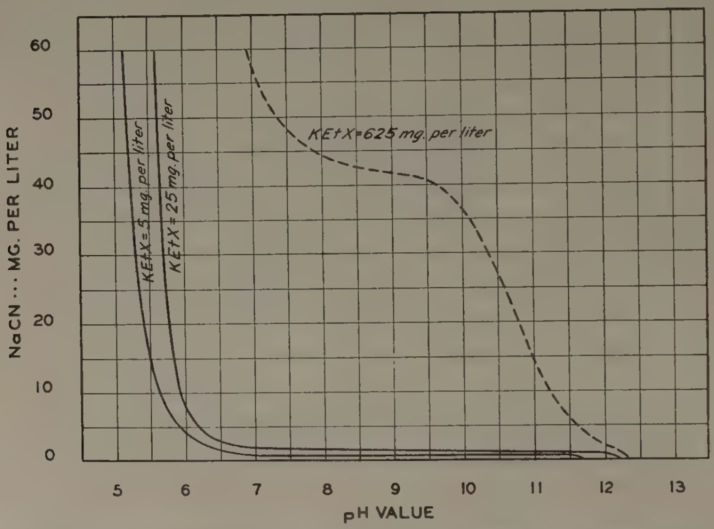

FIG. 8.—INFLUENCE OF CONCENTRATION OF ETHYL XANTHATE IN PRESENCE OF 150 MG. PER LITER $CuSO_4.5H_2O$ ON CONTACT CURVES FOR PYRITE.

Relation to Selective Flotation

Using the data of the preceding sections, a wide range of conditions could easily be specified under which air would effect contact with any given mineral. For differential flotation, however, more exact information is required. Although the curves given above cannot be applied directly to flotation, they can be used to indicate broadly the conditions under which differentiation is greatest. It is again stressed that the concentrations of the reagents added in determining these curves are not changed appreciably by contact with the single mineral specimen used. In the flotation plant, on the other hand, consumption of some of the reagents does occur; consequently their concentrations may be appreciably lower than correspond to the amounts of the reagents added. No direct comparison between the results of the contact tests and actual flotation operations can therefore be attempted until more is known of the consumption of reagents in practice. Other factors too, which have no influence on contact, may influence flotation.

In Australia the separations of most importance are: (1) galena from sphalerite, pyrite and gangue minerals; (2) sphalerite from pyrite and gangue minerals; (3) copper sulfides, e.g. chalcopyrite, from gangue minerals; and (4) pyrite from gangue minerals.

Lead Concentrate.—Because it has no influence on sphalerite, ethyl xanthate seems ideal for the separation of galena from sphalerite, in solutions free from metallic salts and from other collectors. If sphalerite has become activated by copper from the ore a small addition of cyanide should deactivate it (Fig. 3) provided there is not a significant concentration of copper in the pulp. In such a case, and with this reagent combination, deactivation is best carried out within the pH range 8 to 10; below 8 the concentration of cyanide must be increased sharply and above 10 there is a danger that galena may be depressed. These are necessarily inexact limits whose values depend on the xanthate concentration. The xanthate concentration does not require exact control; any unconsumed xanthate will not cause trouble in subsequent operations. The galena-pyrite separation presents greater theoretical difficulties, since a depressant for pyrite is essential. However, because of the greater ease in depressing pyrite than in deactivating sphalerite, it does not present any greater difficulties in practice. Cyanide is a suitable depressant: a small concentration is effective within the pH range 8 to 10. Alkalis alone would not be effective, since galena is affected by them to about the same degree as pyrite. Copper sulfate increases the differentiation between the minerals. In the absence of cyanide the chalcopyrite of the ore will float with the galena. Since the curves for chalcopyrite and activated sphalerite do not differ greatly, it follows that if much more than sufficient cyanide is added to deactivate the sphalerite, the chalcopyrite will also be depressed. In this case it would probably be floated subsequently with sphalerite. The minerals bornite, chalcocite and covellite are all much more difficult to depress by cyanide than chalcopyrite (Figs. 2 and 3, pp. 252 and 253). They should, therefore, float with the galena.

Zinc Concentrate.—The curves for sphalerite (Fig. 5) and pyrite (Fig. 8) suggest that there should be little difficulty in fixing concentrations of xanthate and sodium cyanide that would result in an effective separation of these minerals. However, probably because the activation process is not instantaneous, practical operations do present difficulties of a type not to be foreseen from these curves. Activation is due to copper ions, but in alkaline xanthate solutions most of the copper is precipitated as hydroxide, basic carbonate[6] or xanthate. The very low copper-ion concentration in equilibrium with these salts is nevertheless high enough to activate the sphalerite, but the rate of activation is dependent upon the rate of re-solution of the copper from these precipitates. Corresponding curves for sphalerite and amyl xanthate, presented later, show that less cyanide can be tolerated in the pH region 8 to 10 than elsewhere, and suggest that similar relationships may exist for other collectors, particularly if insufficient time has been allowed for equilibrium to be reached.

The differentiation between sphalerite and pyrite is greater for 150 mg. per liter $CuSO_4.5H_2O$ than for 25 mg. per liter (compare Fig. 4 on p. 253 with Fig. 4 on p. 274). Fig. 6 indicates that there is an optimum xanthate concentration of from 10 to 40 mg. per liter for the sphalerite flotation, and Figs. 5 and 8 indicate that very high xanthate concentrations decrease the differentiation between sphalerite and pyrite.

It would seem to be impossible, using the reagents considered in this paper, to float sphalerite away from chalcopyrite or other copper sulfide minerals. If the chalcopyrite does not float in the lead section, it would be recovered with the zinc. (Interference by factors outside the scope of this paper is not considered.)

Since the common gangue minerals do not respond to ethyl xanthate, even in the presence of copper sulfate, no difficulty should be experienced in floating sphalerite away from them.

Copper Concentrate.—Ethyl xanthate alone does not give satisfactory differentiation between chalcopyrite and pyrite. A depressant for the pyrite is essential, and for this purpose an alkali or cyanide in alkaline solution is generally used. Fig. 3 on page 253 shows that even then the differentiation is not great but Fig. 4 of that paper shows that in the presence of copper sulfate the differentiation is increased enormously. Sufficient copper may dissolve during the grinding of a chalcopyrite-pyrite ore to effect this differentiation.

As previously shown (p. 253), the separation of pyrite from bornite, chalcocite and covellite is more easily effected than its separation from chalcopyrite. In these cases copper sulfate is hardly necessary. The figures in this paper and in the preceding paper show that, with this reagent combination, differentiation between chalcopyrite and sphalerite is possible only over a narrow range of concentrations. Such close control would be difficult to maintain in practice.

Pyrite Concentrate.—With ethyl xanthate or any of the other collectors of this investigation, the separation of pyrite from the gangue minerals with which it is associated should not prove difficult. When cyanide or alkali is present, acidifying and/or addition of extra xanthate should prove satisfactory.

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[6] S. U. Pickering: *Jnl.* Chem. Soc. (1909) **95**, 1409.

POTASSIUM ISO-AMYL XANTHATE

Influence of Alkalis

The influence of the concentration of amyl xanthate on the critical pH values for galena, chalcopyrite and pyrite has not been determined, because all three minerals respond to very low concentrations of the collector, even in alkaline solutions. For sphalerite, however, which responds only to high concentrations, a curve (Fig. 19) has been determined which shows the relationship between the concentration of the collector and the critical pH value. No activator was used. Comparison with Fig. 7 of a paper by Gaudin, Haynes and Haas[7] indicates, in this case, a close agreement between the results from their direct flotation tests and these contact tests.

Influence of Cyanide

Copper-free Solutions.—The influence of cyanide on contact is almost identical for chalcopyrite and pyrite. The curves for 31.6 mg. per liter potassium amyl xanthate showing the relationship between the cyanide concentration and the pH value necessary to prevent contact are given in Fig. 9. Alkali, however, is more effective as a depressant for pyrite, the curve ending at a pH value of 12.3 for pyrite but continuing beyond 13 for chalcopyrite. These curves are lines of approximately constant cyanide-ion concentration, the "constants" being about 2.4 mg. CN per liter for chalcopyrite (variation 4 to 1.5) and 1.7 per liter for pyrite (variation 2.3 to 0.9).

No curve could be obtained for sphalerite under similar conditions, since it does not respond to this concentration of xanthate. The corresponding curve for galena is a straight line parallel to the vertical axis through a pH value of 12.1, the cyanide concentration being without influence on the critical pH value.

Copper-bearing Solutions.—Corresponding curves in the presence of copper sulfate are shown in Fig. 9 for chalcopyrite and pyrite, and in Fig. 10 for sphalerite. The addition of copper sulfate again raises the concentration of sodium cyanide necessary to prevent contact at surfaces of chalcopyrite and preactivated sphalerite, but more than 3NaCN per molecule of copper sulfate must be added. The "flat" portion of the curve (over which the amount of cyanide required to prevent contact does not vary with the pH value) is much more prolonged than when using ethyl xanthate; this is because of the higher critical pH values; i.e., greater tolerance of alkalis when using amyl xanthate. In most cases these critical pH values lie above 13.

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[7] A. M. Gaudin, C. B. Haynes and E. C. Haas: Flotation Fundamentals, pt. IV, 37 (1930).

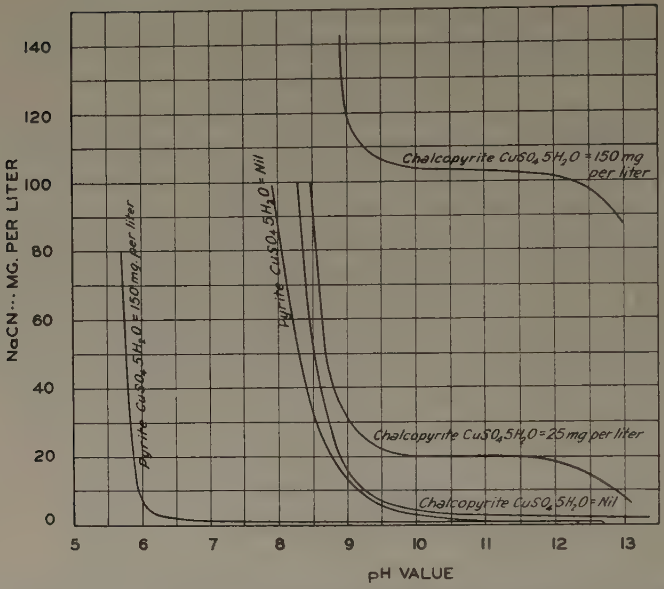

FIG. 9.—CONTACT CURVES FOR CHALCOPYRITE AND PYRITE WITH POTASSIUM AMYL XANTHATE = 31.6 MG. PER LITER, AND INFLUENCE OF COPPER SULFATE THEREON.

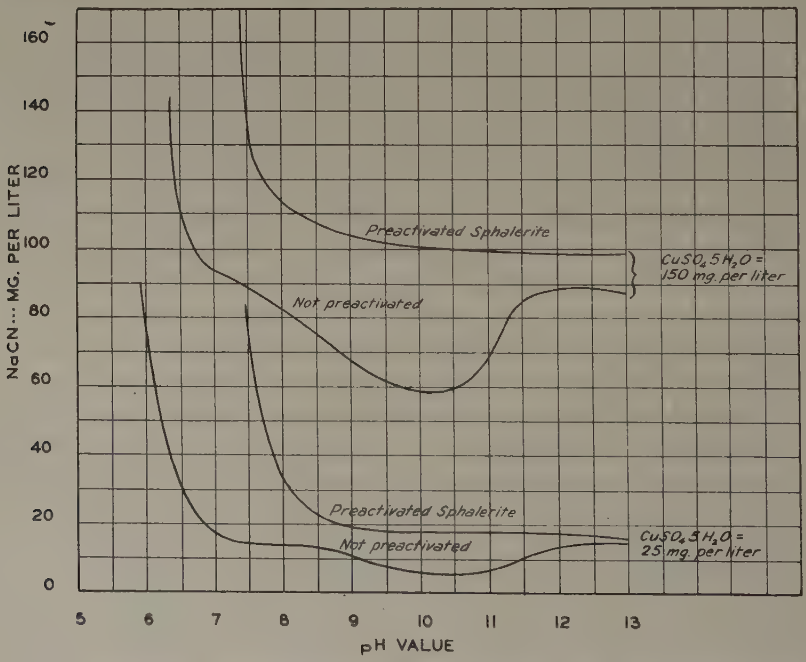

FIG. 10.—INFLUENCE OF CONCENTRATION OF COPPER SULFATE ON CONTACT CURVES FOR SPHALERITE WITH POTASSIUM AMYL XANTHATE = 31.6 MG. PER LITER.

There is included also a composite figure (Fig. 11) that shows how close are the concentrations of cyanide necessary to prevent the formation of a precipitate in the solution and the concentrations of cyanide necessary to prevent contact at surfaces of chalcopyrite and activated sphalerite. This figure should be compared with Fig. 6 on page 254, which shows the corresponding relationships for ethyl xanthate. The concentrations of cyanide required for these three purposes are in closer agreement when using amyl xanthate.

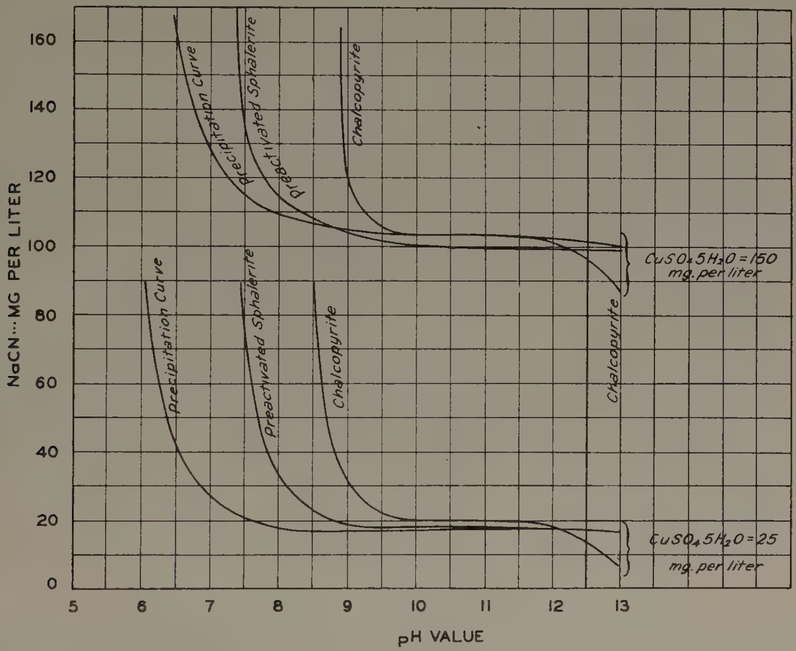

FIG. 11.—COMPARISON OF CURVES SHOWING PRECIPITATION OF COPPER AMYL XANTHATE WITH CONTACT CURVES FOR CHALCOPYRITE AND PREACTIVATED SPHALERITE, WITH POTASSIUM AMYL XANTHATE = 31.6 MG. PER LITER AND: (1) $CuSO_4.5H_2O$ = 25 MG. PER LITER; (2) $CuSO_4.5H_2O$ = 150 MG. PER LITER.

Relation to Selective Flotation

For the separation of galena from sphalerite and pyrite, amyl xanthate has the advantage over ethyl xanthate in that it can be used in more alkaline solutions, in which depression of the other minerals by cyanide is easier. Lime might therefore be substituted for sodium carbonate with a bigger margin of safety. At lower pH values there is the disadvantage that more cyanide would be required to deactivate any accidentally activated sphalerite, and to prevent contact with pyrite.

For the separation of sphalerite from pyrite, because of the dip in the sphalerite curves between pH values of 8 and 11, amyl xanthate is not as effective as ethyl xanthate. For more alkaline solutions, however, amyl xanthate should be better than ethyl xanthate.

Fig. 9 shows that for the chalcopyrite-pyrite separation the presence of copper sulfate, or some similar reagent, is essential. In its absence, there is very little differentiation between the minerals except at high pH values.

Though the contact angle for amyl xanthate (86°) is higher than that for ethyl xanthate (60°) the tenacity of sticking in the latter case is certainly good enough to permit of excellent flotation; no advantage can be claimed therefore for amyl xanthate because of its higher contact angle. Indeed, Ralston and Barker[8] have expressed the view that amyl xanthate leads to special difficulties with the froth, not shared by ethyl xanthate. There is nothing in these results opposed to their suggestion that a mixture should be used, for it would be possible to choose the concentrations of the other reagents in such a manner that the differentiation between any two minerals would not be jeopardized. The curves for amyl xanthate show the nature of the influence of one of the higher xanthates on air-mineral contact, and where it is desired to take advantage of any special virtue shown by them it might be preferable to use butyl xanthate instead. As a collector this would be only slightly less effective than amyl xanthate; it might, moreover, lead to less difficulty with the froth.

SODIUM DI-ETHYL DITHIOCARBAMATE

Influence of Alkalis

The critical pH values for galena and chalcopyrite are very high, being above 13.5 for the standard concentration (26.7 mg. per liter) of the collector. The value for pyrite is 10.5. In Fig. 19 there is shown the relationship between the concentration of the collector and the critical pH value for sphalerite, which responds only to a high concentration of the collector. Owing to the rapid decomposition of the dithiocarbamate in acid solutions, the determination of the curve had to be discontinued at pH 6.5.

Influence of Cyanide

Copper-free Solutions.—Curves showing the relationship between the pH value and the cyanide concentration necessary to prevent contact are given for chalcopyrite (lower curve of Fig. 12) and for pyrite (upper curve of Fig. 13). The chalcopyrite curve is a line of (approximately) constant cyanide-ion concentration, the critical cyanide-ion concentration necessary to prevent contact being 1.8 mg. per liter. Because of the rapid decomposition of the collector in acid solution at pH values below 6.5, the curve for pyrite differs in form from that when using ethyl xanthate. The dithiocarbamate decomposes completely within 15 min. at room temperature at a pH value of 5.5.

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[8] Ralston and Barker: *Trans.* Electrochem. Soc. (1931) **60**, 319.

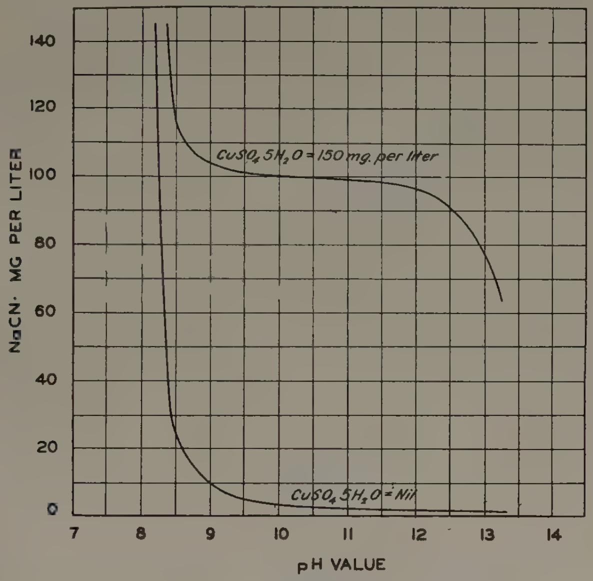

FIG. 12.—CONTACT CURVE FOR CHALCOPYRITE WITH SODIUM DI-ETHYL DITHIOCARBAMATE = 26.7 MG. PER LITER, AND INFLUENCE OF COPPER SULFATE THEREON.

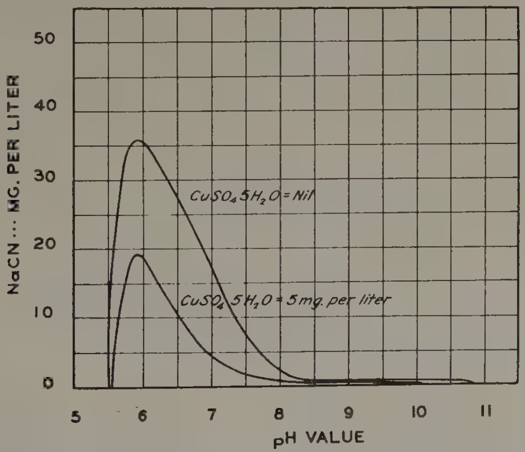

FIG. 13.—CONTACT CURVE FOR PYRITE WITH SODIUM DI-ETHYL DITHIOCARBAMATE = 26.7 MG. PER LITER, AND INFLUENCE OF COPPER SULFATE THEREON.

Copper-bearing Solutions.—The amount of cyanide necessary to prevent contact in the presence of 150 mg. per liter $CuSO_4.5H_2O$ is shown for chalcopyrite in the upper curve of Fig. 12 and for sphalerite and for preactivated sphalerite in Fig. 14. The curves for chalcopyrite and preactivated sphalerite closely resemble those obtained when using ethyl xanthate as collector. The curve for sphalerite not preactivated resembles more closely the curve when using amyl xanthate.

Copper sulfate is extremely active in preventing contact with pyrite. In the presence of 150 mg. per liter $CuSO_4.5H_2O$, no cyanide is required to prevent contact whatever the pH value may be. With 25 mg. per liter contact is possible only at pH values close to 6, and a very small cyanide addition prevents contact.

Even with 5 mg. per liter $CuSO_4.5H_2O$, Fig. 13 shows that very little cyanide is required to prevent contact. Pretreatment of pyrite with copper sulfate followed by washing with distilled water does not influence its subsequent reaction to the dithiocarbamate. Therefore if the pyrite surface is changed by the copper sulfate so that it no longer responds to the collector (which seems unlikely) the reaction must be reversible; i.e., the surface must revert to its original condition on washing with water. Hence it seems impossible to attribute the lack of response of the pyrite to this collector to a simple inhibiting surface film formed by direct reaction with copper sulfate.[9] No cyanide being necessary for depression if sufficient copper sulfate is present, one cannot attribute the depression to cupricyanide ions; likewise alkalis and their reaction products with copper sulfate cannot be responsible. Probably the lack of response

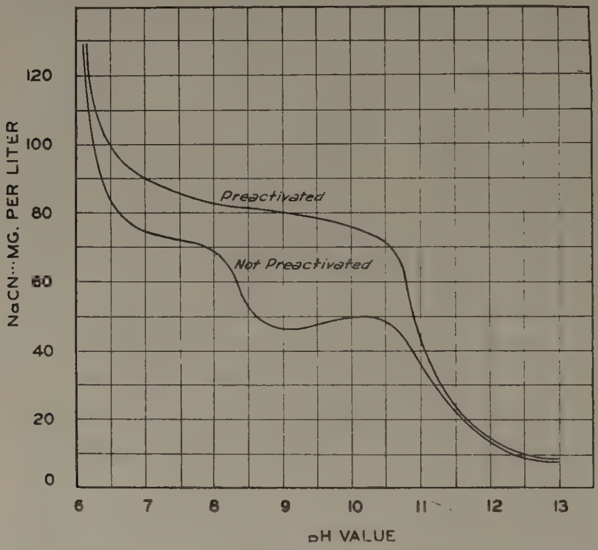

FIG. 14.—CONTACT CURVES FOR SPHALERITE WITH SODIUM DI-ETHYL DITHIOCARBAMATE = 26.7 MG. PER LITER AND $CuSO_4.5H_2O$ = 150 MG. PER LITER.

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[9] E. Berl and B. Schmitt [*Koll. Ztsch.* (1933) **65**, 264] say that weathered pyrite removes no copper from a copper sulfate solution but that freshly ground pyrite removes some copper.

should be attributed to the very low concentration of the collector remaining in solution after precipitation with copper sulfate.[10] (This concentration must, however, be sufficiently great to influence a chalcopyrite surface.) No precipitation curves comparable with those for ethyl or amyl xanthate can be given, since there were precipitates in all the copper-bearing solutions tested. The chalcopyrite curve, however, divides the solutions into two classes with respect to the nature of the precipitates as well as with regard to contact with air. Below the curve, precipitation is immediate and heavy; above the curve, precipitation occurs only after at least 15 min., and is not nearly so dense.

Relation to Selective Flotation

Less rigid control of the alkalinity would be required for the separation of galena from sphalerite and pyrite than when using ethyl xanthate as collector. Lime probably could be used without danger, for the critical pH value of galena with the dithiocarbamate as collector is very high, and at pH values above 11, little cyanide would be required to prevent the flotation of pyrite and to deactivate any sphalerite that had been accidentally activated by copper.

For the sphalerite-pyrite separation the di-ethyl dithiocarbamate possesses the advantage over ethyl xanthate that a lower concentration of copper sulfate suffices to prevent contact at a pyrite surface. Counter to this advantage, but probably not completely annulling it, is the greater difficulty of activating sphalerite in the pH region 8 to 11.

For the chalcopyrite-pyrite separation the dithiocarbamate might be expected to give better results than ethyl xanthate. In the absence of copper sulfate the dithiocarbamate gives slightly better differentiation, and in the presence of copper sulfate the differentiation is increased greatly.

Sodium Di-ethyl Dithiophosphate (Sodium Aerofloat)

We are indebted to the American Cyanamid Co. (through Dr. G. Barsky) for a pure sample of this collector. It is more stable in acid solutions than are the xanthates and dithiocarbamates.

Influence of Alkalis

Fig. 15 shows for galena, pyrite and chalcopyrite the relationship between the concentration of the dithiophosphate and the pH value necessary to prevent contact. It has been shown previously[11] that galena and pyrite do not respond to 25 mg. per liter of the collector in neutral or slightly acid solutions. Their ready response in acid solutions suggests that an examination of the influence of a supposed collector on any given mineral should include tests over a wide range of pH values.

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[10] The concentration of copper sulfate stoichiometrically equivalent to the collector added is 20 mg. per liter; it is not surprising therefore that 25 mg. per liter almost suffices to prevent contact and that 5 mg. per liter, which merely lowers the position of the cyanide pH curve slightly, does not suffice to do so.

[11] Wark and Wark: Reference of footnote 3.

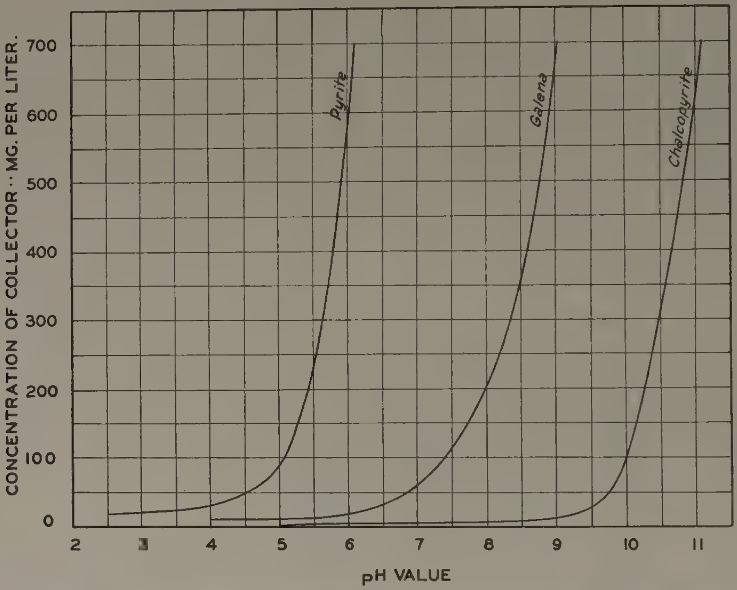

FIG. 15.—RELATION BETWEEN CONCENTRATION OF SODIUM DI-ETHYL DITHIOPHOSPHATE AND CRITICAL pH VALUE FOR PYRITE, GALENA AND CHALCOPYRITE.

Influence of Cyanide

Copper-free Solutions.—Since chalcopyrite alone of the minerals tested responds in neutral or alkaline solutions to 32.5 mg. per liter of the dithiophosphate, the influence of cyanide was determined for that only. The curve (Fig. 20), showing the relationship between the pH value and the concentration of sodium cyanide necessary to prevent contact, corresponds to a critical value of the order 0.01 mg. CN^- per liter. Such a low tolerance of cyanide by chalcopyrite and of alkalis by all the minerals shows that collection by sodium di-ethyl dithiophosphate is more easily prevented by depressants than collection by any other of the collectors referred to in this paper. This may not be a disadvantage, for provided that contact with one mineral can be maintained, the nonresponse of others is desirable.

Copper-bearing Solutions.—Fig. 16 shows for sphalerite, chalcopyrite, pyrite and galena, in the presence of 150 mg. per liter $CuSO_4.5H_2O$, the relationship between the pH value and the concentration of sodium cyanide necessary to prevent contact. The similarity between the curves for sphalerite and galena was expected, since both are primarily activation curves, an activator being required for galena as well as for sphalerite with this collector. The curves show that a lower concentration of copper ions suffices for the activation of galena. With this collector, sphalerite and chalcopyrite both exhibit slightly less tolerance to sodium cyanide than with ethyl xanthate. Tolerance to alkali is also diminished; the "flat" portions of the curves are shortened considerably.

Except in strongly alkaline solutions (pH > 11) the curves for sphalerite and preactivated sphalerite are identical, indicating a speedy activation of the sphalerite. From this point of view the dithiophosphate may have an advantage over ethyl xanthate. There is need for closer control of alkalinity because of the greater sensitivity to alkalis, but the stability of the collector in acid solutions might be an advantage.

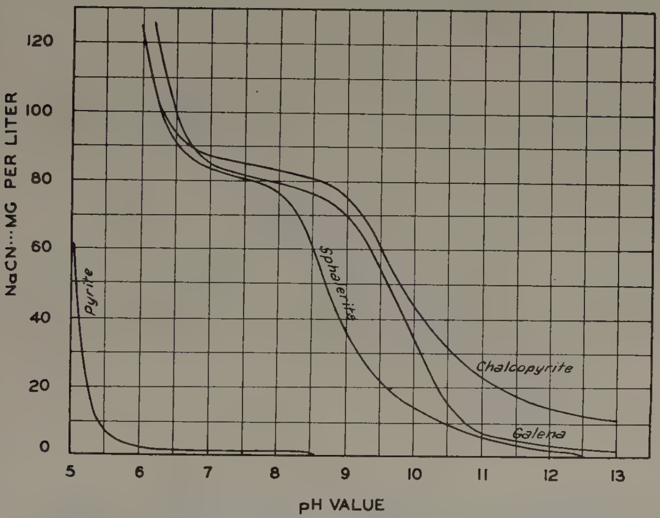

FIG. 16.—CONTACT CURVES FOR CHALCOPYRITE, GALENA, PYRITE AND SPHALERITE WITH SODIUM DI-ETHYL DITHIOPHOSPHATE = 32.5 MG. PER LITER AND $CuSO_4.5H_2O$ = 150 MG. PER LITER.

Pyrite shows, as usual, a marked difference from the other minerals. If a pyrite specimen is first placed in a copper sulfate solution, then washed, and finally placed in a solution of the collector, a bubble of air effects contact with it only after the surface is wiped, and even then the full angle of contact is not attained. Apparently, although copper sulfate can activate pyrite, the activating film is not as permanent as the corresponding film on sphalerite.

Relation to Selective Flotation

In alkaline solutions sodium di-ethyl dithiophosphate would not collect galena unless the mineral was activated. In d saciolutions, however, it could be used for the separation of galena from sphalerite. By careful control of pH value it might even be possible to separate galena from pyrite. Accidental activation of sphalerite or pyrite by heavy metal salts derived from the ore would interfere with the separation and, because of the high concentration that would be required and the danger of using it in acid solutions, cyanide would not be a suitable deactivator.

The reagent could be used to determine the degree to which accidental activation of the galena, pyrite and sphalerite of any given ore had occurred. Any recovery of free particles of these minerals in alkaline solutions would point to activation; provided, of course, that the frother used was not also a collector.

If galena were first removed by another collector, for the reasons just cited, sodium di-ethyl dithiophosphate might offer some advantages for the sphalerite flotation. Though the differentiation between sphalerite and pyrite in the presence of copper sulfate is not as great as when ethyl xanthate is used as collector, it is still sufficiently great for a successful differential flotation.

Sodium di-ethyl dithiophosphate seems very suitable for the chalcopyrite-pyrite separation. In the absence of activators, chalcopyrite responds to a low concentration of this collector, but pyrite does not. In the presence of copper sulfate, which could be added if sufficient were not derived from the ore, the differentiation is as great as when ethyl xanthate is used, in some cases greater.

Potassium Di-n-amyl Dithiocarbamate

Because of their hygroscopic nature, potassium di-n-amyl dithiocarbamate, and the corresponding n-butyl compound proved difficult to prepare. Following the method of Whitby and Matheson,[12] they were formed by the reaction between molecular equivalents of the respective diamines, carbon bisulfide and normal potassium hydroxide solution. Evaporating under reduced pressure on a water bath produced only very viscous liquids. On standing over sulfuric acid in a vacuum desiccator for several days, crystalline solids were obtained. The di-butyl compound can be recrystallized from benzene, or may be purified by dissolving in ethyl ether, adding petroleum ether (boiling point, 40° to 60° C.) and removing the ethyl ether by evaporation. It consists of white crystalline flakes, which soften at about 110° and melt completely at 146° C. (corr.). The di-n-amyl compound is more soluble in all solvents. It was purified by dissolving in ethyl ether, filtering, evaporating and then drying in a desiccator, and finally washing with petroleum ether. The white flaky crystalline product softens at about 110° C. and melts completely at 155° C. (corr.). Both compounds are so hygroscopic that on exposure to air for a few minutes they become liquid.

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[12] Whitby and Matheson: *Trans.* Roy. Soc. Canada (1924) **18**, sec. III, 111.

Influence of Alkalis

Fig. 19 shows the relationship between the pH value and the concentration of the collector necessary to prevent contact with sphalerite. Included in the same figure is the corresponding curve for potassium di-n-butyl dithiocarbamate, which is also a collector for sphalerite in the absence of activators. As might be anticipated with a collector that needs no activator for sphalerite, very high alkalinity is required to prevent contact with the other minerals. For pyrite the critical pH value (with 42.3 mg. per liter of the reagent) is slightly less than 13, and for galena and chalcopyrite the critical pH values are still higher.

Influence of Cyanide

Copper-free Solutions.—Fig. 17 shows the relationship between the pH value and the concentration of sodium cyanide necessary to prevent contact at surfaces of chalcopyrite, pyrite and sphalerite respectively. The curves for sphalerite and pyrite are lines of (approximately) constant cyanide-ion concentration of 6 mg. and 17 mg. CN per liter, respectively. The curve for chalcopyrite is not apparently a line of constant cyanide-ion concentration. It is confined to a more alkaline region than any of the other curves in copper-free solutions, and, as previously

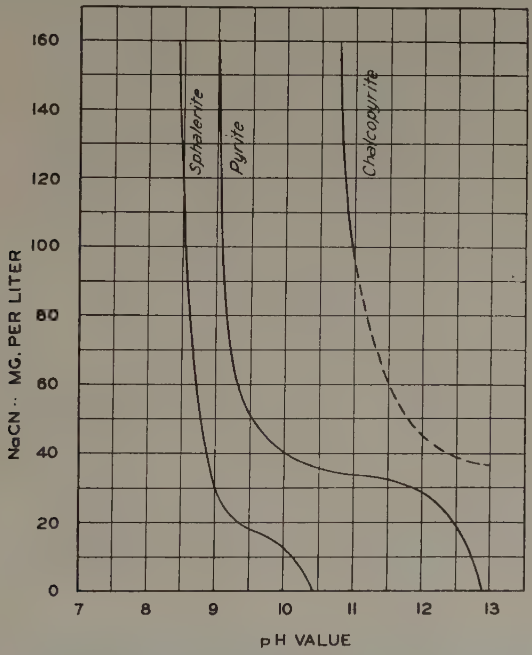

FIG. 17.—CONTACT CURVES FOR CHALCOPYRITE, PYRITE AND SPHALERITE WITH POTASSIUM DI-N-AMYL DITHIOCARBAMATE = 42.3 MG. PER LITER.

stated, its position could not be determined accurately. From the position of the top part of the curve, the critical cyanide-ion concentration is probably of the order of 50 mg. per liter.

The effect of cyanide on contact at a sphalerite surface—noted here for the first time—lends support to the view that cyanide influences contact at surfaces of minerals that form complex cyanides. This effect, however, is hardly what one would have anticipated from the results of Gaudin, Haynes and Haas.[13] These authors state that the flotation of sphalerite caused by a similar compound, di-iso-amyl ammonium di-iso-amyl dithiocarbamate, is not influenced by sodium cyanide. It is surprising that the collecting power of such similar potassium and amine salts should be so differently influenced by cyanide.

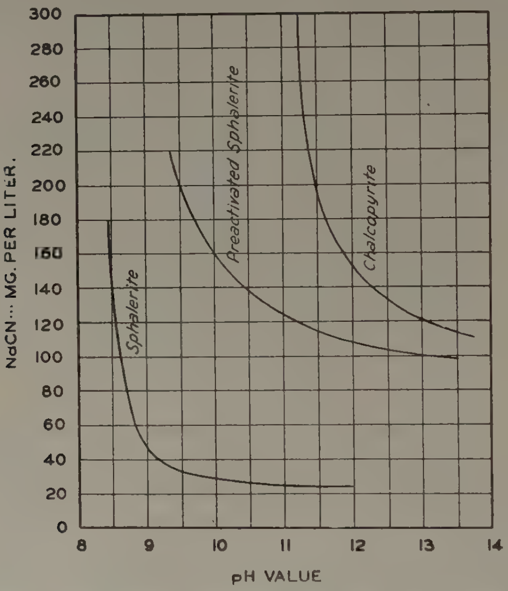

FIG. 18.—CONTACT CURVES FOR CHALCOPYRITE AND SPHALERITE WITH POTASSIUM DI-N-AMYL DITHIOCARBAMATE = 42.3 MG. PER LITER AND $CuSO_4.5H_2O$ = 150 MG. PER LITER.

Copper-bearing Solutions.—Fig. 18 shows for fresh sphalerite, for sphalerite that has been previously activated by copper sulfate, and for chalcopyrite (each in the presence of 150 mg. per liter $CuSO_4.5H_2O$) the relationship between the pH value and the concentration of sodium cyanide necessary to prevent contact. The two curves for sphalerite differ more widely than the similar curves with the other collectors.

The low position of the curve for sphalerite that had not been preactivated is presumably due, for the more strongly alkaline solutions, to the precipitation of most of the copper by the collector and depressants, thus preventing activation in a region where activation is necessary. Both curves for sphalerite rise sooner on the left than the similar curves for the other collectors. This is to be attributed to the fact that sufficient cyanide must be added both to deactivate the sphalerite, and, at pH values below 10.4, to prevent contact at the unchanged sphalerite surface (Fig. 19). A higher concentration of cyanide is required to prevent contact at a chalcopyrite surface than with the other collectors, which was to be expected, since in the absence of copper sulfate a higher concentration is also required.

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[13] Gaudin, Haynes and Haas: Reference of footnote 7.

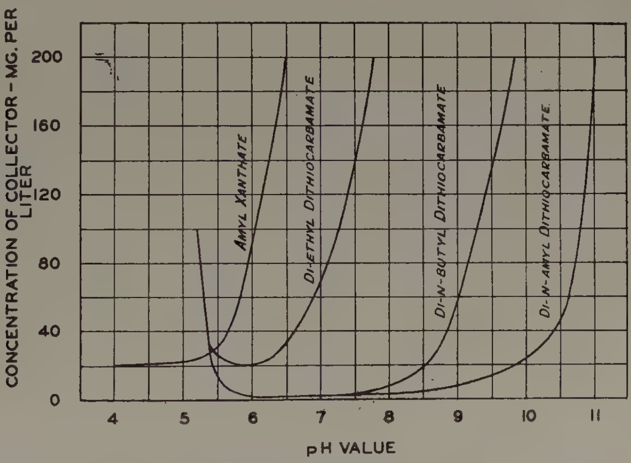

FIG. 19.—RELATION BETWEEN CONCENTRATIONS OF VARIOUS COLLECTORS AND CRITICAL pH VALUE FOR SPHALERITE.

The corresponding curve for pyrite could not be determined, since 150 mg. per liter $CuSO_4.5H_2O$ completely prevents contact when using this collector at a concentration of 42.3 mg. per liter. At any pH value between 6.6 and 13 no cyanide is required to prevent contact. The influence of copper sulfate when using the di-n-amyl dithiocarbamate is, in fact, similar to that when using the di-ethyl dithiocarbamate (see p. 286). If cyanide is also added, between 80 and 150 mg. per liter, it apparently combines with part of the copper, and there then remains sufficient of the collector to be adsorbed by the pyrite; in no such case, however, was the full angle of contact observed.

Relation to Selective Flotation

Differentiation between galena and the other minerals when using this collector demands much higher concentrations of cyanide and/or alkali than are necessary for ethyl xanthate. Separation of galena from sphalerite would be entirely dependent on the use of a depressant for the latter mineral. If cyanide were used for this purpose, the collector would not be as suitable for the subsequent flotation of sphalerite as is ethyl xanthate, because the mineral would not respond as readily to copper sulfate. On the other hand, chalcopyrite responds very readily to the collector, both in the presence and in the absence of copper sulfate. To obtain the maximum differentiation between this mineral and pyrite, the presence of copper sulfate is again desirable, since it practically prevents the response of the pyrite.

GENERAL DISCUSSION

Although the di-amyl dithiocarbamate leads to a high contact angle and the minerals are responsive to a lower concentration of it than of the other collectors used, it does not appear to offer any advantages for selective flotation over them. Indeed, it is probably inferior to them, because of the need to add more of the depressants required to produce differentiation between the minerals. The use of collectors of which an exceedingly low concentration produces a response in the sulfide minerals does not appear to be attractive, for, as with the di-amyl dithiocarbamate, differentiation would be difficult to control. Nor does the use of collectors to which the sulfide minerals are appreciably less sensitive than they are to sodium di-ethyl dithiophosphate appear attractive. Apparently there is an intermediate range of compounds from which collectors should be chosen. Among the sulfur-bearing polar-nonpolar compounds, those that form heavy metal salts of the same order of solubility as the ethyl xanthates are most useful.

There follows for each of the four minerals considered a comparison of the results given by all five collectors.

Chalcopyrite

Of the four minerals tested, chalcopyrite responds most readily to soluble sulfur-bearing collectors. The ability of the depressants to prevent contact with air is shown in Table 3.

TABLE 3.—*Concentrations of Depressants Necessary to Prevent Contact at a Chalcopyrite Surface*

Collector	Concentration of Collector, Mg. per Liter	Critical pH Value	Critical CN^- Concentration, Mg. per Liter
Sodium di-ethyl dithiophosphate	32.5	9.6	(0.01)
Potassium ethyl xanthate	25	11.8	0.4
Sodium di-ethyl dithiocarbamate	26.7	>13.5	1.8
Potassium iso-amyl xanthate	31.6	>13	2.4
Potassium di-n-amyl dithiocarbamate	42.3	>13.5	(50) approx.

In the presence of copper sulfate there is an increase in the amount of cyanide necessary to prevent contact; differences of the same order remain between the collectors, but in terms of the total added cyanide they are relatively smaller. The additional cyanide required presumably converts the added copper to one of the complex copper cyanides. Copper sulfate causes a corresponding increase in the concentration of alkalis necessary to prevent contact; this is manifested in the displacement of the curves to the right.

Galena

Galena and pyrite respond with about equal readiness to the type of collector considered in this paper, both being less responsive than chalcopyrite and more responsive than sphalerite.

Adsorption of the collector by galena being unaffected by cyanide, the differences between the critical pH values for corresponding concentrations of the collectors serve best to indicate the differences between the collectors. The critical pH values for the standard concentrations are 6.5 for the dithiophosphate, 10.4 for ethyl xanthate; >13.5 for the di-ethyl dithiocarbamate, 12.1 for amyl xanthate, and >13.5 for the di-n-amyl dithiocarbamate.

Pyrite

The effects of cyanide and of alkalis on the adsorption of the collectors by pyrite are summarized in Table 4.

TABLE 4.—*Concentrations of Depressants Necessary to Prevent Contact at a Pyrite Surface*

Collector	Concentration of Collector, Mg. per Liter	Critical pH Value	Critical CN^- Concentration, Mg. per Liter
Sodium di-ethyl dithiophosphate	32.5	4.1	
Potassium ethyl xanthate	25	10.5	0.1
Sodium di-ethyl dithiocarbamate	26.7	10.5	
Potassium iso-amyl xanthate	31.6	12.3	1.7
Potassium di-n-amyl dithiocarbamate	42.3	12.9	17

The results obtained for pyrite in the presence of copper sulfate have not yet been satisfactorily interpreted. In seeking an explanation several diverse facts must be borne in mind:

1. Except in acid solutions pyrite does not respond without activation to sodium di-ethyl dithiophosphate. Its response in the presence of copper sulfate indicates that copper sulfate can activate pyrite.

2. The critical pH value for pyrite in the presence of 25 mg. per liter potassium ethyl xanthate is raised from 10.5 to above 12 by 150 mg. per liter $CuSO_4.5H_2O$. There is also an increase when using amyl xanthate. Activation must occur in both cases.

3. Pyrite responds, without activation, to sodium di-ethyl dithiocarbamate, but 150 mg. per liter $CuSO_4.5H_2O$ prevents the response. Fig. 13 shows that 5 mg. per liter does not completely prevent the response. The relationships with potassium di-n-amyl dithiocarbamate are similar. The copper salts of the dithiocarbamates are exceedingly insoluble, however, and one is tempted to suggest that the copper sulfate added reduces the concentration of the dithiocarbamate ion to such a low value that no dithiocarbamate is adsorbed.

4. When both cyanide and copper are present, it seems probable that the cupricyanide ion is the effective depressant.

Sphalerite

Of the collectors tried (in the standard equivalent concentrations) potassium di-n-amyl dithiocarbamate alone is adsorbed from neutral solutions by sphalerite in the absence of an activator. With higher concentrations or in acid solutions sphalerite responds to amyl xanthate and to di-ethyl dithiocarbamate. The critical pH values for the usual concentrations are 5.5 for amyl xanthate, approximately 6.3 for the di-ethyl dithiocarbamate, and 10.4 for the di-n-amyl dithiocarbamate. Cyanide is an effective depressant even in the absence of copper sulfate.

Sphalerite, when activated by copper sulfate, responds to both collectors and activators in a manner and to an extent quantitatively very similar to chalcopyrite. The activated sphalerite is slightly more sensitive to depressants. Except with the dithiophosphate, cyanide is more effective in preventing activation of sphalerite than in de-activating sphalerite already activated. That is, in the presence of copper sulfate and a collector, less cyanide is required to prevent contact with sphalerite than to destroy the contact with sphalerite previously activated by copper sulfate. The difference between the amounts of cyanide necessary for fresh sphalerite and for preactivated sphalerite is negligible for di-ethyl dithiophosphate, small for ethyl xanthate, rather more important for the di-ethyl dithiocarbamate and amyl xanthate, and greatest for the di-n-amyl dithiocarbamate. The difference is greatest between pH values of 8 and 11.

With some ores cyanide is not as effective in preventing the flotation of blende or marmatite with the lead concentrate as the figures would imply. Perhaps some of the blende may be carried into the froth mechanically, but some at least probably floats because of activation by heavy metal salts other than copper salts. It is a matter of importance to determine the nature of this supposed activation. A possible

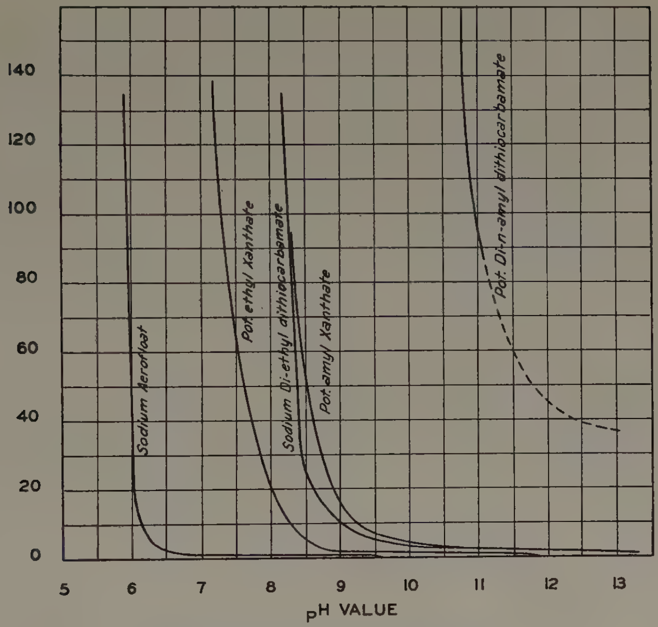

FIG. 20.—CONTACT CURVES FOR CHALCOPYRITE WITH STANDARD EQUIVALENT CONCENTRATIONS OF VARIOUS COLLECTORS.

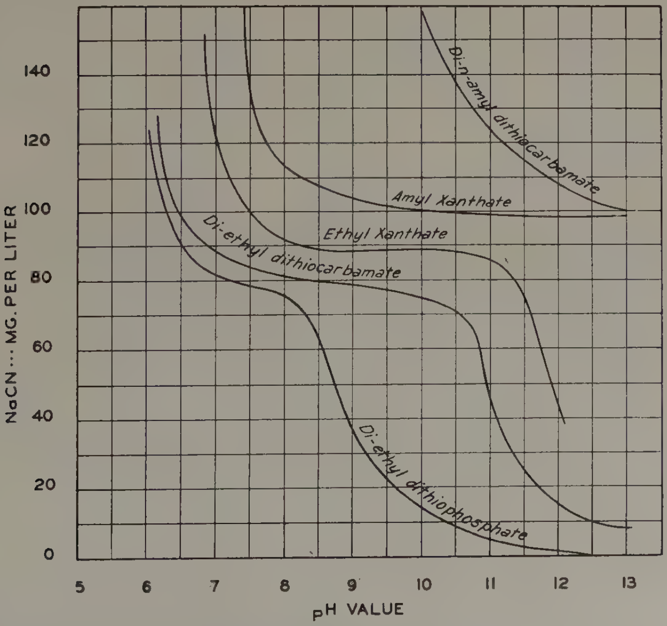

FIG. 21.—CONTACT CURVES FOR SPHALERITE, PREVIOUSLY ACTIVATED BY COPPER SULFATE, WITH STANDARD EQUIVALENT CONCENTRATIONS OF VARIOUS COLLECTORS AND $CuSO_4.5H_2O$ = 150 MG. PER LITER.

activator is dissolved lead; this would be unaffected by cyanide. Berl and Schmitt[14] have shown that sphalerite removes lead from a solution of its salts. The rarer constituents of the ore may be responsible.

There are three possible types of interpretation for the positions of the curves for sphalerite. These positions may be determined by the amount of cyanide necessary to dissolve or prevent the formation of: (1) the activating copper-bearing film, (2) a film consisting of xanthate adsorbed by the activating film, or (3) the xanthate film, leaving the activating film unchanged. The possibility must be borne in mind that one curve may owe its position to one of these actions, another curve to another action.

Sphalerite specimens, removed from solutions in which they have failed to respond to xanthate because of a too high ratio of cyanide to copper sulfate, have been washed and placed in xanthate solution. They have not then responded, therefore it seems that action 3 is excluded. Fig. 21 shows that the concentration of cyanide necessary to prevent contact at an activated sphalerite surface is dependent upon the particular collector employed; this fact indicates that action 1 does not afford the correct explanation in this case. Additional evidence leading to the same conclusion has been obtained from direct tests in which the amount of cyanide necessary to dissolve dual copper and xanthate films from sphalerite has been determined. The quantity is dependent upon which particular xanthate is used. This is further support for the view that the copper-bearing film is more easily removed and its formation more easily prevented than the dual copper and xanthate film. Any accidental activation of sphalerite by copper salts would therefore be more easily removed before the addition of the collector.

Significance of Critical pH Value

Barsky[15] noted a relationship between the critical pH value and the concentration of ethyl xanthate necessary to induce contact at a galena surface; namely, that the product of the hydrogen-ion concentration and the xanthate-ion concentration is approximately constant. Similar approximate constancy holds in alkaline solutions for the corresponding curves for other minerals and other collectors, but in acid solutions the concentration of the collector is invariably greater than corresponds with this relationship. It is not possible to decide whether this is due to the inadequate nature of the relationship, for agreement in acid solutions would be prevented by such disturbing factors as: (1) consumption of the collector, (2) formation of the free acid corresponding to the collector, (3) decomposition of the collector. The loss of collector ions would be relatively more important than in alkaline solutions because of the lower concentration required by the relationship. The case most nearly conforming to the Barsky relationship is that of sphalerite and potassium di-butyl dithiocarbamate, the figures for which are given in Table 5. In Table 6 are given the figures for galena and sodium di-ethyl dithiophosphate, the case in which the variation is greatest. The variation in the value of the product is already pronounced in alkaline solutions and becomes still greater in acid solutions. While the evidence cannot be regarded as conclusive, there is in the other cases as good agreement as in the original case of galena and ethyl xanthate. As already stated in reply to Dr. Barsky, our interpretation is that the concentration of alkali necessary to prevent adsorption of a collector by any mineral is directly proportional to the concentration of the collector. This result is in agreement with both physical and chemical theories of the mechanism of the adsorption.

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[14] Berl and Schmitt: Reference of footnote 9.

[15] Page 236, this volume.

TABLE 5.—*Sphalerite and Potassium Di-n-butyl Dithiocarbamate*

Concentration of Collector, Mg. per Liter	Critical pH Value	Product (H^+) (Collector Ion$^-$)
200	9.8	1.3×10^{-13}
100	9.3	2.1×10^{-13}
50	8.9	2.6×10^{-13}
20	8.5	2.6×10^{-13}
10	8.2	2.7×10^{-13}
5	7.8	3.3×10^{-13}
2	7.3	4.1×10^{-13}

TABLE 6.—*Galena and Sodium Di-ethyl Dithiophosphate*

Concentration of Collector, Mg. per Liter	Critical pH Value	Product (H^+) (Collector Ion$^-$)
700	9.0	3.4×10^{-12}
400	8.6	4.8×10^{-12}
200	8.0	9.6×10^{-12}
100	7.4	19×10^{-12}
50	6.9	30×10^{-12}
25	6.3	(60×10^{-12})
10	5.5	(150×10^{-12})

SUMMARY

The study of the influence of alkalis, cyanide and copper sulfate on contact between an air bubble and the minerals galena, sphalitere, pyrite and chalcopyrite respectively, has been continued. Five different soluble sulfur-bearing collectors, including members of three different

organic types, have been used to induce contact. The main conclusions from the investigation, apart from those enumerated on page 265 are:

1. Qualitatively, the effects of alkalis, cyanide and copper sulfate are substantially the same for all five collectors. The quantitative measures of the effects differ according to the collector used.

2. With regard to their power to resist the action of depressants, the five collectors investigated may be placed in the following order, the greatest resistance being shown by the last mentioned: (1) sodium-di-ethyl dithiophosphate (sodium Aerofloat), (2) potassium ethyl xanthate, (3) sodium di-ethyl dithiocarbamate, (4) potassium amyl xanthate, (5) potassium di-n-amyl dithiocarbamate. This is the inverse order of the solubility of the heavy metal salts formed from the collectors.

3. If the concentrations of alkalis, cyanide and copper sulfate were properly controlled, any collector of these types could be used, apparently, for the separations required with sulfide ores.

4. The prevention of air-mineral contact by cyanide in alkaline solution is due to the prevention of the adsorption by the mineral of a film of collector. It is not due to the formation of a cyanide film at the surface.

5. The optimum relative alkali, cyanide and copper sulfate concentrations when using each of the five specified collectors are considered for the following separations: (1) galena from sphalerite, pyrite and gangue minerals, (2) sphalerite from pyrite and gangue minerals, (3) chalcopyrite from pyrite and gangue minerals, (4) pyrite from gangue minerals.

These considerations are based on differences in air-mineral contact, but they should serve to indicate broadly the conditions where differentiation is best in practical flotation. Absolute coincidence is not to be expected. Certainly, with ethyl xanthate, these relative concentrations agree reasonably well with the additions found most satisfactory in practice. Bearing this limitation in mind, it appears that for certain separations some of the collectors may possess metallurgical (but not necessarily economic) advantages over ethyl xanthate; for example, sodium di-ethyl dithiophosphate for the production of copper and of zinc concentrates, and sodium di-ethyl dithiocarbamate for the production of copper concentrates. On the other hand, amyl xanthate and the di-n-amyl dithiocarbamate do not seem to offer any advantages compared with ethyl xanthate, except possibly to separate sphalerite from gangue minerals, but not from pyrite, without the use of an activator.

6. Particular attention has been devoted to the question of the concentration of the collector when using ethyl xanthate. It has been found that there is no apparent advantage in increasing the concentration above 15 mg. per liter. Indeed, for the flotation of sphalerite, high concentrations would be harmful. (These remarks apply only for

concentrations. Because of the consumption of reagents, concentrations cannot be calculated directly from the additions made in practice.)

7. Chalcopyrite responds to the selected standard equivalent concentrations of all five collectors. The amount of cyanide or alkalis necessary to prevent contact with the different collectors increases in the order cited under 2. Critical pH values and critical cyanide-ion concentrations are given in Table 3. If copper sulfate is present, more cyanide or alkali is required to prevent contact, the additional cyanide being proportional to the amount of copper added.

8. Galena responds to the standard equivalent concentrations of all the collectors except sodium di-ethyl dithiophosphate. In the presence of copper sulfate it responds, like sphalerite, to this collector. Contact with galena not activated in this manner is uninfluenced by cyanide but is influenced by alkalis.

9. Like galena, pyrite responds to all the collectors except sodium di-ethyl dithiophosphate. With the several collectors cited, cyanide and alkalis are effective in preventing contact in the order cited under 2. Critical pH values and critical cyanide-ion concentrations are given in Table 4. The influence of copper sulfate is complex. Thus, with sodium di-ethyl dithiophosphate under certain conditions, it can activate pyrite; with the dithiocarbamates, in certain solutions, it can prevent contact with pyrite; finally, in conjunction with cyanide and alkali, it can prevent contact with pyrite, with any of the five collectors.

10. Sphalerite responds in neutral solutions to the standard equivalent concentration of only one of the collectors, the di-n-amyl dithiocarbamate. Cyanide and/or alkali prevent contact in this case. Activation is necessary to induce contact when using the other collectors. If copper sulfate is used, activation in the presence of a collector may be difficult within an intermediate pH range (8 to 11 in these contact tests) the difficulty of activation (as estimated by the amount of cyanide that can be tolerated) increasing as the series cited in 2 above is descended.

11. In the presence of a collector, less cyanide is required to prevent activation of fresh sphalerite than to deactivate sphalerite that has been previously activated by copper. The respective amounts approach each other as the series cited in 2 is ascended, and become identical when the di-ethyl dithiophosphate is used. More cyanide is necessary to remove a dual copper-xanthate film from a sphalerite surface than to remove the copper-bearing activation film.

12. The relationship noted by Barsky between concentration of ethyl xanthate and the critical pH value necessary to prevent contact at a galena surface can be extended to other collectors and other minerals with approximate accuracy. This is interpreted as implying that the concentration of alkali necessary to prevent adsorption is proportional to the concentration of the collector.

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Hypothesis for the Nonflotation of Sulfide Minerals of Near-colloidal Size

By A. M. Gaudin,* Member A.I.M.E. and Plato Malozemoff,* Junior Member A.I.M.E.

In modern practice the major portion of an ore is reduced to flotation size by fine grinding. As the result of grinding fine enough to liberate most of the minerals, a substantial portion (generally 5 to 15 per cent) of the ore is inadvertently reduced to a size much finer than that necessary for liberation. This portion of most flotation pulps is very refractory to recovery by flotation. A reduction of one-quarter or more in recovery in this range of extremely fine particles can be observed. The loss in metals caused by overgrinding is appreciable. That this loss is characteristic of very fine particles has been suspected for some time;(1)† however, it was definitely quantified only recently.(2) Definite reduction in floatability with reduction in particle size beyond a critical size of maximum floatability can be accepted now as an established fact.

Formulating the Hypothesis

In looking for the cause of the ill behavior of the finest particles it may be well to consider the essential differences between fine and coarse particles. A difference that immediately comes to mind is their intrinsic difference in size and consequently in relative extent of surface. If one agrees that attachment of mineral particles to bubbles must follow direct encounter of particles with bubbles one is forced to conclude(3) that the probability of encounter between a mineral particle and a bubble varies directly as the size of the particle, provided the particle is small compared to the bubble. In other words, the finer the particle the poorer its chance of being recovered in a given time of flotation.

There is an effect, implicit in this theory, which should overcome the effect of excessive fineness of the particles; namely, flocculation of the particles. The floccules can be conceived as being simply larger particles and therefore capable of becoming attached to bubbles. And, indeed, in all the cases in which a sulfide mineral of near-colloidal size was successfully floated the mineral (but not necessarily the gangue) was flocculated. Moreover, it was shown experimentally(4) that galena previously flocculated by inorganic flocculators and then added to gangue

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† References are at the end of the paper.

minerals could be separated from the latter by customary flotation methods, whereas galena previously dispersed by inorganic deflocculators and then added to gangue minerals could not be separated satisfactorily in that fashion.

Another difference between the fine mineral particles and the coarse, which perhaps is not so obvious, is the fact that fine particles have a surface older on the average than coarse particles. Once a very fine particle is produced in a comminuting operation the chances are very great that it will remain of that size until most of the solids are also reduced to that size. This is the well-known "protective" action of coarse particles(5) in crushing and grinding. Since near-colloidal particles are of a size considerably finer than that to which the bulk of the ore is commonly ground in practice, it is not likely that they will be further ground, so long as coarser particles are present. The fine particles, therefore, fail to acquire a constantly refreshed surface. On the other hand, the coarser particles, comprising the bulk of the solids in the pulp, have their surface constantly rejuvenated in the process of being reduced in size by grinding, so that when they are reduced to the requisite size and are removed from the grinder-classifier circuit their surfaces are considerably younger than the surfaces of the finest particles.

It is entirely probable that an old mineral surface is different from one that is freshly opened by crushing: all sorts of chemical alterations may take place while a surface remains in contact with a complex aqueous solution, so that an old surface may fail to react with a collector upon the belated addition of the latter (as in flotation circuit). Because the extent of chemical alteration is a function of time, it is reasonable to expect that under the conditions of laboratory batch-grinding tests, as well as in practice, the older mineral surfaces are more profoundly altered than the younger. Thus the fine particles, having a surface older on the average than coarser particles, will behave differently.

Chemical alteration of older mineral surfaces could be due to the chemical agencies present in the grinding atmosphere and capable of dissolving in the pulp solution, and/or to those derived from the solid surfaces present in the system and dissolved by the pulp solution.

To the first class of agencies belong oxygen and carbon dioxide, assuming for the moment that the remaining constituents of the air are inert. Oxygen, dissolved in water, acts on the freshly broken sulfide surfaces to form some sort of oxidation coatings.(6) Carbon dioxide gives rise to carbonate ions, which may form an insoluble carbonate coating on the coating on the surface of the sulfide mineral by reaction with the oxide or suboxide formed through the agency of oxygen. However, both these effects, the formation of carbonate coatings and of oxidation coatings, should not affect adversely the flotation by the common collectors of lead and copper minerals, since the compounds of these metals composing such coatings react readily with the customary collectors to form heteropolar insoluble compounds which, if properly oriented, will effect flotation.

To the second class of agencies belong such compounds as are dissolved by the pulp solution from the gangue and from the grinding surfaces (balls or pebbles, and mill walls).

Recently Guido R. M. del Giudice postulated(7) that the gangue slime-coatings, which he observed to form on galena under certain conditions, are attached to the galena by a cement formed as the result of a metathetical reaction between the cation of the galena, Pb^{++}, and the anion of the gangue; for example, a cement of lead carbonate in the case of a calcite gangue. According to del Giudice, the formation of such a cement is conditioned by the insolubility of the compound of which the cement is composed. Moreover, he believes that the slime-coatings on some galena particles are responsible for their nonflotation.

Following the same general reasoning, it can be postulated that poor recovery by flotation of sulfide mineral particles will result, even in the absence of slime-coatings, if the surface of these particles simply become coated with a compound consisting of the anion of the gangue and the cation of the mineral. This will take place provided an opportunity is given for the formation of such a depressing coating *before* the collector is permitted to form its own coating on the mineral surface. Both the collecting coating and the depressing coating are extremely insoluble, so that equilibrium conditions are hardly approached, even after long conditioning. Thus time is the salient *practical* factor in the problem, even though in the hypothetical case in which equilibrium is attained the most important factors are those of concentration of the collector and of anions derived from the gangue, and of solubility and ionization of the respective coatings.

Poor flotation would result under the conditions outlined above, irrespective of the size of the mineral particles, because size is not a factor in these conditions. In practice, however, chiefly the fine particles are affected because, as it has already been explained, they have more time to be affected by the chemical agencies. In finely ground pulps, moreover, the action is likely to have proceeded further because the average distance between particles is smaller, so that ion transfer is necessarily facilitated.

The two explanations of the phenomenon of poor flotation of near-colloidal particles are, of course, not exclusive of each other. Fine particles produced in grinding that have a young surface and pass immediately into the flotation circuit are probably adversely affected by the mechanical cause of poor flotation of fine particles: that of noncollision of the particles with bubbles; while the chemical cause in addition to the mechanical cause is simultaneously operating on particles whose surface is old; and finally, the chemical cause is operating more intensely and quickly, the finer the average size of the particles in the pulp.

EXPERIMENTAL PROCEDURE

In the investigation of the problem of flotation of minerals of near-colloidal size, which have hitherto been believed substantially unfloatable, an experimental procedure had to be employed that would reduce the number of variables to a minimum. Accordingly, mixtures of a pure sulfide mineral with gangue were ground with water in porcelain jars (Abbé pebble mills, "assay" size) with flint pebbles long enough (21 hr.) to reduce all of the mineral mixture to a state of subdivision that can be termed near-colloidal. Examination under the microscope of the ground product revealed that the largest particles in the pulp were 3 microns in diameter, whereas the average size of the *visible* particles was about 1 micron. A large proportion of the pulp was in a state of subdivision too fine to be seen under the microscope. On the basis of specific surface produced, the average size of the particles is estimated from the data given above at about one-third micron.

Investigation was confined to four of the commonest mineral sulfides (galena, chalcocite, chalcopyrite and pyrite) and to three gangues: granite (Butte quartz monzonite containing chiefly plagioclase of intermediate composition, orthoclase, and quartz, together with some biotite, hornblende, magnetite and titanite), aplite (also from Butte, consisting mainly of quartz, orthoclase and a little plagioclase), and limestone (relatively pure marble).

The ground charges were floated in a 500-gram Fahrenwald flotation machine. The flotation products thus obtained, concentrate and tailing, were analyzed for the metal of the sulfide mineral. From the analyses of the products and their weight, recoveries and selectivity indices (a precise measure of the effectiveness of flotation) were calculated. The relation between these and the arbitrary variables furnished the data from which conclusions of a general nature, embodied in this paper, were drawn.

Potassium n-amyl xanthate was used as the collector. In some of the tests, especially in those in which a very small amount of collector was used, the froth formed was insufficient for rapid flotation. In such tests enough terpineol was added to induce a normal rate of froth overflow. Otherwise no frother was added. Terpineol does not appear to affect the effectiveness of flotation as measured by selectivity indices, but only its rate.

EXPERIMENTAL RESULTS

It was found that if the collector is added into the pebble mill and is present during grinding, the agency that causes poor flotation of the fine particles is almost completely overcome and effective flotation of the sulfide mineral results. This discovery conforms nicely with the hypotheses proposed in this paper to explain the poor flotation of slime particles formed by grinding in the absence of collector. The chemical cause and the mechanical cause for poor flotation of these particles are simultaneously overcome. The inert coatings are prevented from forming because the collector coating forms first. Once this coating is formed, the surface of the particles becomes nonpolar and relatively stable; flocculation immediately ensues because the surface is of a vastly different polarity than the water, or, from another viewpoint, because the charges on the surface of the mineral particles that kept them in a dispersed state are no longer present. As the result of flocculation, the fine particles no longer behave individually but as floccules; that is, as larger particles that are capable of colliding readily with air bubbles.

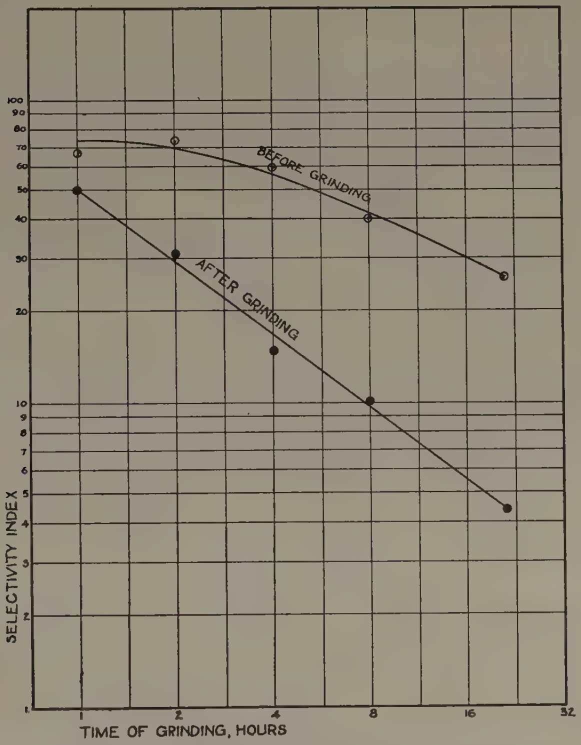

FIG. 1.—EFFECT OF PLACE OF ADDITION OF REAGENT ON FLOTATION OF PROGRESSIVELY FINER PULPS OF GALENA-APLITE MIXTURES.

Improvement in the flotation of even relatively coarse pulps (maximum particle size, 30 microns) can be observed if the reagent is added into the pebble mill. This, be it noted, agrees with plant observations.(8) Table 1 and Fig. 1 show the effect of the place of addition of the reagent on flotation of progressively finer pulps of galena-aplite mixtures.

TABLE 1.—*Effect of Place of Addition of Reagent on Flotation of Progressively Finer Pulps of Galena-aplite Mixtures*

Time of Grind, Hr.	Potassium Amyl Xanthate, Lb. per Ton.		Weight of Concentrate, Per Cent	Grade, Per Cent Pb		Selectivity Index	Recovery Per Cent
	Added before Grind	Added after Grind		Concentrate	Tailing		
1	0.57		23.2	69.8	0.08	68	99.62
2	1.14		22.9	70.6	0.073	74	99.65
4	2.3		24.4	64.	0.073	58	99.65
8	4.6		26.1	59.6	0.11	41	99.48
21	12.			47.[a]	0.15[a]	25	99.3[a]
1		0.57	22.6	69.2	0.14	50	99.31
2		1.14	18.7	67.6	0.33	31	97.92
4		2.3	25.5	58.6	0.84	14.8	95.98
8		4.6	29.9	49.9	1.13	10.2	94.96
21		12.		45.[a]	4.5[a]	4.5[a]	80[a]

[a] Obtained by interpolation from the data for Fig. 2 and Table 2 for the amount 12.0 lb. potassium amyl xanthate per ton.

Lacking actual experimental data on the relation of time of grinding and the resulting size of the ground product, Rittinger's law[(9)] was invoked by assuming that the amount of surface produced is directly proportional to the time of grinding. This can be assumed because the original surface is negligible compared with the surface produced after grinding. Thus the amount of the reagent necessary to cover that surface would also be

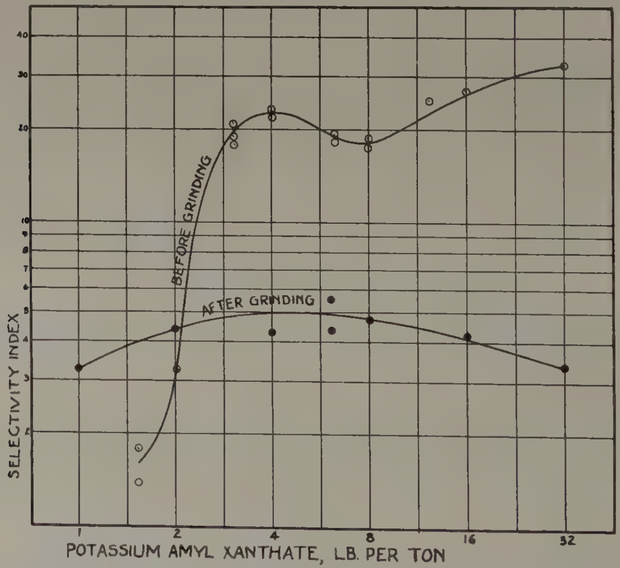

FIG. 2.—EFFECT OF INCREASING AMOUNTS OF POTASSIUM N-AMYL XANTHATE ON FLOTATION OF GALENA-APLITE MIXTURES ADDED BEFORE OR AFTER GRINDING.

directly proportional to the time. It can thus be considered that in this series of tests there is only one arbitrary variable, time of grinding (an inverse function of the size of particles).

In the following series of tests the time of grinding was kept constant (21 hr.), the arbitrary variable being the amount of the reagent added. The data are presented in Table 2 and Fig. 2.

TABLE 2.—*Effect of Increasing Amounts of Potassium Amyl Xanthate on Flotation of Galena-aplite Mixtures Added Before and After Grinding*

Potassium Amyl Xanthate, Lb. per Ton.		Weight of Concentrate, Per Cent	Grade, Per Cent Pb		Selectivity Index	Average Selectivity Index for Duplicate Tests	Recovery, Per Cent
Added before Grind	Added after Grind		Concentrate	Tailing			
1.43		11.3	24.6	14.4	1.4	1.6	17.9
1.43		8.2	34.	14.2	1.8		17.5
2.		27.6	39.	5.92	3.3		71.7
2.86		28.7	50.2	0.39	17.5	19.2	98.10
2.86		29.8	51.3	0.35	19.		98.41
2.86		33.6	44.7	0.21	21.		99.08
4.		32.9	45.9	0.20	22.	22.5	99.12
4.		33.2	45.4	0.18	23.		99.21
5.7		32.7	46.2	0.30	18.3	18.5	98.68
5.7		34.2	44.4	0.26	18.7		98.89
8.		32.2	45.	0.30	17.7	18.	98.61
8.		30.8	46.2	0.30	18.2		98.56
11.4		31.3	47.2	0.17	25.		99.21
16.		32.9	56.	0.22	27.		99.20
32.		31.6	56.6	0.16	33.		99.23
	1.	43.9	31.9	4.54	3.3		84.6
	2.	27.6	45.8	4.75	4.4		78.3
	4.	23.6	46.7	5.26	4.3		78.3
	5.7	37.4	36.9	3.05	4.4	5.	87.4
	5.7	30.5	44.2	2.80	5.6		87.2
	8.	28.3	47.7	4.55	4.7		80.4
	16.	31.3	42.6	4.65	4.2		80.6
	32.	40.8	32.7	4.35	3.4		83.7

It is obvious from Table 2 and Fig. 2 that flotation of galena when the reagent is added before grinding is greatly superior to that when the reagent is added after grinding. The curious shape of the before-grinding curve was totally unexpected and is as yet unexplained. However, a curve of similar shape in flotation of chalcocite-aplite mixtures is explained elsewhere(10) on the basis of different reaction products formed at the different points of maxima and minima on the curve. It is significant that in the range of small amounts of reagent, if the reagent is added before grinding, results are poorer than if the reagent is added after grinding.

In addition to potassium amyl xanthate, other collectors(11) produce effective flotation on a number of sulfide minerals when the collectors are added into the pebble mill before grinding, so as to be present while the mineral is ground.

Testing the Hypothesis

Oxygen is always present in a grinding system and will cause the formation of oxidation coatings on the surface of the sulfide mineral particles. Although Taggart, Taylor and Knoll(6) believe that these oxidation coatings are not only beneficial but necessary for flotation of sulfide minerals, a certain adverse effect can be observed that can be ascribed to the presence of oxygen during grinding. This becomes evident by comparing the series of tests just presented with parallel tests in which the minerals were ground in the virtual absence of oxygen, and the results of which are presented in Table 3 and Fig. 3. In these tests oxygen was eliminated from the pebble mill by sweeping the mill atmosphere free of air, alternately introducing nitrogen and evacuating. The mineral mixture was ground in a partial vacuum after two flushings with nitrogen. The amount of oxygen present during grinding can thus be reduced to a very small figure (of the order of magnitude of 10^{-4} mol). Flotation was carried on in air as usual.

Table 3.—*Effect of Increasing Amounts of Potassium Amyl Xanthate on Flotation of Galena-aplite Mixtures Ground in Absence of Air*

Potassium Amyl Xanthate, Lb. per Ton, Added before Grind	Weight of Concentrate, Per Cent	Grade, Per Cent Pb		Selectivity Index	Recovery, Per Cent
		Concentrate	Tailing		
0.57		no flotation		1	
1.		no flotation		1	
2.	25.5	58.9	0.30	25	98.54
4.	29.4	53.6	0.21	26	99.06
8.	28.7	55.8	0.18	30	99.20
16.	28.	54.9	0.18	29	99.17
32.	27.3	58.1	0.19	33	99.24

Comparison of Table 3 and Fig. 3 with Table 2 and Fig. 2 shows that when the pulp is ground in the absence of oxygen a much smaller amount of collector is sufficient to yield certain results than that necessary to yield the same results when the pulp is ground in air (620 mm.). The relative consumption of the collector in the two cases could have been predicted from the theory of Taggart and his associates that the collector reacts with oxidation coatings on mineral surfaces. The reason for the improvement in the flotation of minerals when they are ground in absence of oxygen is not at all clear; yet this improvement shows that oxygen is a factor that can by no means be neglected.

However, that oxygen is not responsible for the subnormal behavior of sulfides becomes evident in comparing the following tests on flotation of galena-aplite mixtures, which were ground under various conditions. Also, they show that the time of contact of gangue and galena has a profound influence on the behavior of galena in flotation, irrespective of the size to which galena is ground. The results obtained are presented in Table 4.

In Test No. 1 all the agencies that may occur during grinding to cause poor flotation of galena were eliminated, except air. Galena was coarsely ground, but agitated for the standard time of 21 hr., so that whatever influence time alone could have exerted was permitted to take effect. The mineral and the gangue were ground separately, so as to come in contact only in the flotation cell. Flotation was good. On the other hand, in test No. 2 definitely poorer results were obtained: galena was ground for a short time with the gangue, just before floating. The size to which the mineral was ground in tests 1 and 2 is roughly comparable because the gangue in test 2 offered little or no resistance to grinding: it was reduced to near-colloidal size before grinding of galena commenced. And test 3 shows that increasing the time of contact between the gangue and galena, without changing any other conditions, is enough to cause the subnormal behavior of galena in flotation that is observed in a pulp that is much finer and that is prepared and floated in the standard way (test 4).

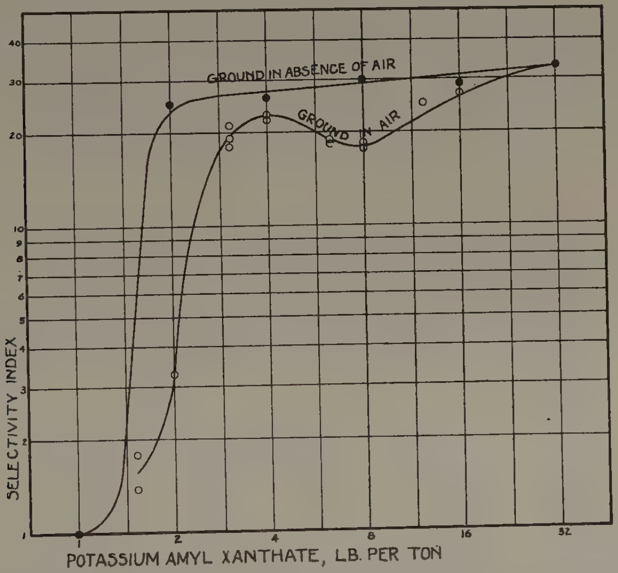

FIG. 3.—EFFECT OF INCREASING AMOUNT OF POTASSIUM AMYL XANTHATE ON FLOTATION OF GALENA-APLITE MIXTURES GROUND IN AIR AND IN ABSENCE OF AIR.

Tests 5 and 6 again demonstrate the influence of time of contact between galena and gangue before floating under otherwise identical conditions. In spite of the larger amount of xanthate used in test 6, in which the time of contact between mineral and gangue is greater, flotation is much inferior to that of test 5.

Test 1.—Standard amounts of galena and aplite (70 and 280 grams respectively) were ground separately. The galena was ground for 1 hr., then the pebbles were removed from the mill and the mineral was agitated in the closed mill for 20 hr. longer; aplite was ground for 21 hr. After the 21 hr. the contents of the two pebble mills were combined and floated with 1.72 lb. potassium amyl xanthate per ton (0.3 gram).

Test 2.—A standard amount of aplite was ground for 20 hr., then galena was added to the pebble mill and the mixture was ground for 1 hr. longer. The charge was then floated with 1.72 lb. potassium amyl xanthate per ton. Thus the relatively coarsely ground galena was floated in presence of aplite slime, but it was ground with the slime only for 1 hr.

Test 3.—Test 2 was repeated, except that after grinding the mixture the pebbles were removed and the mixture agitated in the closed mill for 21 hr. longer before floating.

Test 4.—A standard charge of galena and aplite was ground for 21 hr. and floated with 5.7 lb. potassium amyl xanthate per ton (1.0 gram).

Test 5.—A standard charge of galena and aplite was ground for 1 hr. and floated with 0.57 lb. potassium amyl xanthate per ton (0.1 gram).

Test 6.—A standard charge of galena and aplite was ground for 1 hr., the pebbles were then removed and the mixture was agitated in a closed mill for 20 hr. longer. The charge was floated with 1.72 lb. potassium amyl xanthate per ton.

TABLE 4.—*Effect of Various Conditions in Grinding on Flotation of Galena-aplite Mixtures*

Test No.	Potassium Amyl Xanthate, Lb. per Ton. Added after Grind	Weight of Concentrate, Per Cent	Grade, Per Cent Pb		Selectivity Index	Recovery, Per Cent
			Concentrate	Tailing		
1	1.72	23.3	58.6	0.15	35.	99.62
2	1.72	25.2	59.0	0.48	19.6	97.61
3	1.72	19.3	60.9	9.53	4.4	60.4
4	5.7	37.4	36.9	3.05	4.4	87.4
5	0.57	22.6	69.2	0.14	50.0	99.31
6	1.72	22.9	66.9	0.95	17.7	95.44

It may be remarked in connection with the tests in this series that inhibition of the mineral by the gangue is more marked when the pulp is more finely ground; that is, when the distance of particles from each other is lessened. In the outline of the hypothesis given earlier, it was postulated that the reason for the poor flotation of near-colloidal pulps was perhaps that the fine particles remained long enough in the pulp to have their surfaces coated by inert compounds of the cation of the mineral and of anion of the gangue. The behavior of galena in the tests presented in Table 4 is readily explained in that way. Tests 7 to 12 give further weight to the hypothesis. In tests 10, 11 and 12 the calcite-galena mixtures were ground 14 hr. instead of 21 hr. (standard time for aplite-galena grinds) to arbitrarily allow for the relatively greater softness of the calcite. The results obtained for this series of tests are presented in Table 5.

Test 7.—Test 4 was repeated except that 2 lb. sodium silicate (water glass) per ton was added to the pebble mill before grinding.

Test 8.—Test 1 was repeated in every detail except that 14.3 lb. sodium silicate per ton was added to the galena before grinding.

Test 9.—Test 1 was repeated in every detail except that 14.3 lb. sodium carbonate per ton was added to the galena before grinding.

Test 10.—A standard charge of galena and calcite was ground for 14 hr. and floated with 5.7 lb. potassium amyl xanthate per ton.

Test 11.—Test 10 was repeated except that 4 lb. sodium silicate per ton was added into the pebble mill before grinding.

Test 12.—Test 10 was repeated except that 14.3 lb. sodium silicate per ton was added to the pebble mill before grinding.

TABLE 5.—*Effect of Some Anions on the Flotation of Galena*

Test No.	Potassium Amyl Xanthate, Lb. per Ton. Added After Grind	Weight of Concentrate, Per Cent	Grade, Per Cent Pb		Selectivity Index	Recovery, Per Cent
			Concentrate	Tailing		
7	5.7	33.9	34.5	5.85	3.	75.1
8	1.72	2.8	14.9	12.90	1.2	3.2
9	1.72	25.3	54.9	0.34	21.	98.21
10	5.7	53.4	31.7	0.12	20.	99.66
11	5.7	18.0	23.6	16.15	1.3	24.0
12[a]	5.7	12.0	20.	13.75	1.3	16.5

[a] Just before the end of the flotation operation excess of xanthate (11.4 lb. per ton) was added to the pulp, but no improvement in the operation was observed.

Let tests 2, 4, 7 and 8 be considered in the light of the concentration of silicate ion present. If these tests are enumerated in the order of the amount of silicate ion present they should probably be arranged in the following way: 2 and 4, then 7 and 8. In the first two the silicate anions are naturally furnished by the dissolution of the gangue. In tests 7 and 8 the concentration of silicate ion is artificially increased. It is apparent from the flotation results of these tests that effectiveness of flotation varies as some inverse function of the concentration of silicate ion. In applying this conclusion to practice it can be said that the most adverse effect from a silicate gangue would be expected from gangues that slime badly—because they present an enormous surface for dissolution—and from those that naturally are more soluble.

But the concentration of silicate ions is not the only factor that prevents flotation of galena by forming on its surface an inert coating (possibly of lead silicate). As has already been pointed out, time—that is, time allowed for the approaching of the equilibrium in the reaction between Pb^{++} and the silicate ion—is of paramount significance. To show this it is sufficient merely to compare tests 2 and 6. Although the concentration of silicate ion was possibly greater in test 2, because the aplite is more finely ground and hence presents more surface for dissolution, so that saturation of the solution is more nearly approached, yet flotation in test 6 is poorer because the condition of chemical equilibrium between the smaller concentration of silicate ions and Pb^{++} was more nearly reached, owing to longer time of contact of the galena with the solution containing these silicate anions.

It is also noteworthy that in test No. 8 galena was ground only 1 hr. and is of as floatable a size as galena of test No. 1; and yet no flotation was obtained in test 8. This seems to substantiate the hypothesis that the inert coatings would prevent the flotation of particles irrespective of their size. Also, slime-coatings as such could hardly have been responsible for the nonflotation of the galena particles in test 8 because no gangue slime was present with the galena before the contents of the two pebble mills were combined, and yet no flotation resulted of a sample of this galena when a collector was added to it at that time.

Test No. 9 shows that the carbonate anion does not form an inert coating capable of affecting appreciably the floatability of galena. In test 10 carbonate ion is naturally furnished by the dissolution of the gangue, calcite. Here again, as in test 9, flotation of galena is not adversely affected. Yet if aplite, say, were used in place of calcite and the test conducted under exactly identical conditions, the poor results shown by test No. 4 would again be obtained. The results of test No. 10 would have been better than they were if so much calcite had not floated. The fact that the gangue did float leads one to the very interesting speculation that this may have been because a coating of lead carbonate had formed on the surface of calcite particles. In other words, the hypothesis set forth in this paper—viz., that the surface of the sulfide mineral is made inert by ions migrating to it from the gangue—may have a counterpart: gangue minerals may be activated by metal ions from the sulfide mineral. The conditioning factors in such activation are, of course, solubility of the oxidation coating on galena, solubility of the gangue, and the limitations imposed by the law of mass action. (Another possible explanation for the abnormal presence of gangue in the concentrate may be that the gangue was entrained there as slime-coatings on galena; but the amount involved appears too large for this explanation to hold, and furthermore, according to del Giudice, sulfides coated with slimed gangue should fail to float.)

The inhibiting action of silicate ions on galena is again demonstrated by tests 11 and 12.

Some tests were made using sphalerite (ZnS) as a gangue together with galena. It was found impossible to depress the sphalerite: that mineral floated together with the galena, which behaved in somewhat subnormal fashion. Since there was no copper present to activate the sphalerite, and because potassium cyanide (the usual depressant for sphalerite) seemed totally useless in depressing the sphalerite, it would seem that the mineral was activated owing to migration of Pb^{++} ions and their precipitation at the surface of sphalerite as some insoluble compound. This observation lends further weight to the assumption that nonflotation of sulfide mineral particles of near-colloidal size results because the surfaces of very fine particles of gangue and mineral gradually become identical.

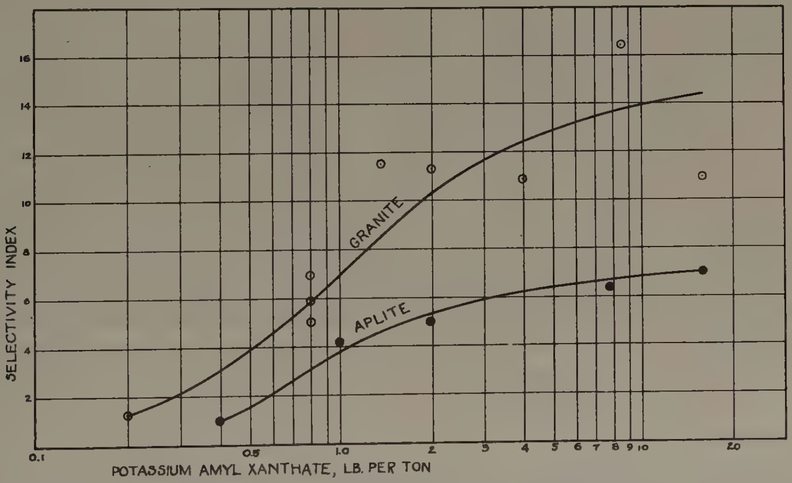

FIG. 4.—EFFECT OF APLITE AND GRANITE GANGUE ON FLOTATION OF CHALCOCITE WITH POTASSIUM N-AMYL XANTHATE ADDED AFTER GRINDING.

Comparison of the effect of granite and aplite gangue on the flotation of chalcocite with potassium amyl xanthate added after grinding into the flotation machine is afforded by Table 6 and Fig. 4.

The difference in the flotation of chalcocite when ground with these two gangues can probably be attributed to the difference in concentration of the silicate ions yielded by the two rocks in water. Aplite contains more quartz, also its feldspar is alkali feldspar, which should yield more silicate in alkaline solution than does the calcium feldspar which predominates in a quartz monzonite. Thus aplite yields more silicate ions into solution than does the granite, hence its presence inhibits flotation of chalcocite more than does the presence of granite. These observations may help explain the value of lime in flotation: its use reduces the con-

TABLE 6.—*Effect of Aplite and Granite Gangue on Flotation of Chalcocite with Potassium Amyl Xanthate Added after Grinding*

Kind of Gangue	Potassium Amyl Xanthate, Lb. per Ton	Grade, Per Cent Cu		Selectivity Index	Recovery, Per Cent
		Concentrate	Tailing		
Aplite	0.4	28.5	10.5	1.9	29.1
	1.	32.4	3.1	4.2	85.8
	2.	30.	1.91	5.	90.8
	8.	31.	1.27	6.4	94.3
	16.	33.1	1.15	7.1	95.5
Granite	0.2	19.2	12.2	1.3	14.3
	0.4	38.	1.94	6.	90.6
	0.8	34.1	1.68	5.9	92.1
	0.8	32.4	2.21	4.9	98.9
	0.8	36.8	1.42	6.9	93.1
	1.4	38.4	0.52	11.7	97.6
	2.	42.7	0.71	11.3	96.4
	4.	35.6	0.70	10.8	97.2
	8.5	39.3	0.29	16.4	98.6
	17.	28.9	0.32	11.9	99.0

centration of silicate ion by "closing" the surfaces of the siliceous minerals to solution.

SUMMARY AND CONCLUSIONS

It seems very probable that one essential reason for poor flotation of the extremely fine portion of flotation pulps is the fact that a large part of that portion is composed of particles with old surfaces which, owing to their age and to the presence of anions derived from the gangue, are rendered unfloatable by the formation of coatings, composed of the cation of the sulfide mineral and the anion of the gangue, that are inert to the action of the collector. The likelihood of formation of such coatings is conditioned by the insolubility of the compound of which they are made and the time allowed for the chemical equilibrium to be approached.

This hypothesis does not hold for particles that are fine but whose surfaces nevertheless are young. Here the mechanical cause for poor flotation of fine particles applies: that of low probability of collision of the particles with bubbles.

An interesting generalization can be drawn from the chemical hypothesis of inert coatings. One can conceive of a pulp in the process of grinding as a system that is subject to continuous chemical changes, obeying in these changes the chemical laws. It seems that in a system composed of different mineral species, and therefore having different free surface-energy levels, changes take place that will tend to equalize these different energy levels and alter the mineral surfaces accordingly. Thus, in the relatively simple system of galena and aplite gangue the surface of galena is so altered as to form a surface that possesses free surface energy comparable to that of the gangue: namely, the formation, presumably, of lead silicate coating; or again, in the system of galena and calcite gangue the formation, presumably, of lead carbonate coating on calcite.

Applying further the hypothesis here formulated, it becomes clear why the so-called primary slimes and "slimy" oxidized ores are so refractory to treatment by flotation. They are composed essentially of the fine particles that have had their surface exposed for a long time to the action of salts in the pulp so that their surface has become coated by an inert compound. Since, owing to their size, they do not become ground while the coarse particles of the ore go through the processes of comminution, their behavior in flotation is naturally subnormal.

Poor flotation of sulfide minerals owing to the formation of inert coatings takes place irrespective of the size of sulfide mineral particles, provided exposure to the causes of this poor flotation is prolonged sufficiently.

The formation of inert coatings, so injurious to flotation, can be partly prevented by restricting the concentration of the anion forming it. The formation of these coatings can be most completely prevented by the addition of the collector into the grinding machine so that the collector shall be present in the grinding system while the mineral particles are being broken and expose their fresh surfaces. In this way the collector is permitted to cover the surface of the mineral first and thus protect it against other agencies in the system.

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Reactions of Xanthates with Sulfide Minerals

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Xanthates have been used as collectors of sulfide minerals in the concentration of ores by flotation for over a decade, yet much remains to be done to elucidate the underlying reasons for the remarkable effectiveness of these compounds in this connection. Briefly stated, the known facts concerning the effect of these reagents on the floatability of minerals are as follows:

1. Xanthates are without effect on silicates, silica, and generally on minerals currently regarded as "gangues."
2. Xanthates in very small amount increase enormously the floatability of sulfides of copper, lead, silver, iron and mercury.
3. If zinc sulfides are first "activated," as by treatment with a copper salt, they are readily floated by xanthates; otherwise not.
4. Elemental copper, silver and gold have their floatabilities increased by xanthates.
5. Sulfides of elements that are not typically metallic—e.g., stibnite, realgar, molybdenite—are not made to float markedly better by treatment with xanthates.
6. Oxidized minerals of lead and copper are floated if treated with large amounts of xanthates, but not so if treated with amounts of the same order of magnitude as required for sulfide flotation. Oxidized zinc minerals are not floated unless xanthates having unusually long hydrocarbon chains (e.g., eight carbon atoms or more) are used. Iron oxides and carbonate are not floated by xanthates.

From a flotation standpoint the most striking feature about xanthates is the very small amount required to accomplish startling changes in the behavior of some minerals. The amounts used in practice to effect excellent concentration are frequently of the order of 0.05 lb. of agent per ton of ore; that is, of the order of one part of agent per forty thousand parts of ore. Changes in amount of agent of the order of 0.01 lb. per ton of ore (or 0.002 lb. per ton of pulp, on a 4:1 basis) have marked effects on the metallurgical results obtained. Yet these changes represent a variation of but one part per million parts of pulp.

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ADSORPTION THEORIES

The extraordinary effectiveness of the xanthates for sulfide flotation appeared, and still appears, explainable only as a surface phenomenon. Preliminary estimates were therefore made of the surface of mineral available for reaction with the xanthates, and of the thickness of the xanthate film that would obtain if all the reagent were spread evenly over that area. These estimates showed that the xanthate filming frequently was one molecule thick.(1)* Taken together with the heteropolar character of xanthate molecules, and with the apparently changed polarity of the mineral following treatment with a xanthate solution, it was postulated that the xanthate molecules (or ions) were adsorbed as a monomolecular film at the surface of the mineral, the xanthate molecules being oriented with their hydrocarbon chains away from the mineral in the same way as adsorbed phenol molecules were thought to be oriented at the surface of galena.(2) When subsequent experiments showed that there was abstraction of xanthate ion from solution by the mineral treated but no abstraction by minerals such as quartz, feldspar, or calcite, which were not floated, the hypothesis gained increased probability of being correct.

However, the accordance between fact and theory was not as good as might have been wished. In the first place, lengthening the chain of the hydrocarbon part of the molecule, which would have made the monomolecular film thicker, necessitated the use of less collector rather than more, for effective flotation. Conversely, shortening of the hydrocarbon chain to a length shorter than that of the ethyl group necessitated a much greater quantity of xanthate, and, furthermore, was relatively ineffective in floating coarse mineral particles. So the theory had to be modified to allow for the formation of a partially complete monomolecular film, flotation being regarded as resulting not only when the filming was complete but, indeed, from an incomplete film, the necessary completeness of film being a function of the length of the hydrocarbon chain and of the size of the particles to be floated.(3)

In many cases the amount of reagent required to effect flotation appeared to exceed considerably that necessary to film monomolecularly the particles to be floated. Since, furthermore, the agent was "used up" by the mineral, a discrepancy appeared between theory and fact. Fortunately for the theory, just about that time Gross and Zimmerley's investigations of crushing and grinding(4) showed that there was in crushed materials much hidden surface in the form of cracks and crevices, so that the general form of the theory could be retained.

Experiments conducted in 1928–29 in the laboratory of one of the writers showed that xanthate abstraction could be considerably in excess of that required to form a monomolecular film, at least in the case of pyrite, provided enough xanthate had been present in the aqueous solution. Of course much less xanthate than that was sufficient to cause flotation, but patently reaction between mineral and agent could go beyond the monomolecular stage. This, of course, made the whole matter of Langmuirian adsorption appear less probable. Furthermore, the question arose as to what was the adsorbed entity if adsorption was the *modus operandi* of xanthate action. The adsorbed entity could be the alkali xanthate molecule, the xanthate ion, the xanthic acid molecule (obtained by hydrolysis), a base-metal xanthate molecule, or some oxidation product derived from the xanthate ion.

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* References are at the end of the paper.

REACTION BETWEEN OXIDIZED MINERALS AND XANTHATES

Again, the fact that xanthate ions are readily abstracted by malachite, azurite, cerussite and anglesite, among other minerals,(5) indicated that contrary to earlier beliefs(3) some action occurred between oxidized minerals and xanthates. Coatings of base-metal xanthates on these treated minerals were observed and photographed.(6) In one instance(7a) the coating produced on cerussite by potassium n-amyl xanthate was extracted by leaching the mineral with acetone, and the leach liquor was evaporated. The residue was identified microscopically and chemically as lead n-amyl xanthate. By treatment with a barium chloride solution the aqueous solution from which the xanthate had been abstracted was found to contain carbonate ion in an amount equivalent to the xanthate abstracted. This can be regarded as absolute proof of the metathetical reaction:

$$PbCO_3 + 2[ROC(S)S]^- \rightarrow Pb[ROC(S)S]_2 + CO_3^{--} \qquad [1]$$

In a further series of tests one-gram portions of crushed cerussite sized to 100 to 600 mesh by screening and sedimentation were treated with 50 c.c. of aqueous potassium n-amyl xanthate solutions of various concentrations at 20° C. for 10-min. periods. Following this treatment the mineral suspension was poured over a filter; an aliquot part of the filtrate was titrated for xanthate ion with iodine after bringing the solution to pH 6 to 7 by addition of dilute acid; the residue on the filter was washed with water, then added in a glass-stoppered bottle to a two-phase system consisting of petroleum ether and a saturated aqueous solution of potassium iodide. Titration was made with a petroleum ether solution of iodine.(8) This gave the amount of xanthate as lead xanthate on the cerussite. The results obtained are presented in Table 1.

These results show that with a reaction time of 10 min. the abstraction of xanthate from solution is practically complete if the amount of xanthate is not excessive, but that if a very large amount of xanthate is present (e.g., over 30 lb. per ton of mineral) the extent to which the reaction proceeds is almost independent of the concentration of xanthate in solution. This shows that the coating of lead xanthate is sufficiently compact to prevent further reaction, or else that the concentration of carbonate ion in solution becomes sufficient to drive reaction 1 from right to left. By increasing the time of reaction ninefold, to 90 min., a substantial but by no means proportionate increase in xanthate abstraction is noted. Thus a duplication of test No. 7 save for an increase in the time of reaction to 90 min. gave an abstraction of 12.81 mg. instead of 10.20 mg. Likewise, increasing the temperature from 20° to 50° C. results in an increase in xanthate abstraction for a 10-min. reaction time from 10.20 mg. to 13.03 mg.

TABLE 1.—*Removal of Potassium n-amyl Xanthate from Aqueous Solution (50 c.c.) by Cerussite (1 Gram)*

Test No.	Potassium n-amyl Xanthate Added, Mg.	Potassium Xanthate Left in Solution, Mg.	Potassium Xanthate Removed by Cerussite (as Lead Xanthate), Mg.	Discrepancy, Mg.
1	2.69	0.17	2.59	+0.07
2	7.01	0.17	6.93	+0.09
3	8.08	0.31	7.78	+0.01
4	10.66	2.13	8.50	−0.03
5	11.13	2.40	8.74	+0.01
6	17.06	6.78	10.26	−0.02
7	21.30	11.10	10.20	0.00
8	27.70	17.00	10.55	−0.15
9	53.25		10.98	
			Average discrepancy	0.05

These facts suggest that the coating formed must be thick. This is substantiated by what estimates can be made of the amount of agent required to form a monomolecular film. Assuming the mineral used to have an average size of 50 microns and a surface three times as large as cubes of the same average diameter (this is intended to allow for irregularity in shape, cracks and crevices(4)), a close-packed monomolecular layer of amyl xanthate ions or lead xanthate molecules having a length estimated at 7Å. (7 × 10^{-8} cm.) would require about 0.04 mg. of potassium xanthate. The film of lead xanthate that can form readily on cerussite in 10 min. is then some several hundred molecules or ions thick, on the average.

CHEMICAL-REACTION THEORY

In view of the known oxidizability of sulfide minerals(9) it was postulated by Taggart, Taylor and Knoll(10) that oxidation of the sulfides precedes their being made floatable by xanthate treatment, and that such a treatment results in the formation of a film of xanthate of the base metal on the sulfides. Oxidation of the sulfides cannot be regarded as proceeding uniformly, since there is considerable evidence that many reactions involving two phases do not proceed evenly at the phase boundary,(11) so oxidation and hence the "xanthating" of sulfide minerals may well be spotty.

There remains, of course, to identify ions given up to the solution by the treated mineral(12) and to ascertain whether the coating actually is the base-metal xanthate.

STUDY OF COATINGS ON TREATED SULFIDE MINERALS

A study has been made at Columbia University of the solution in which galena had been exposed to a xanthate. Complete proof of the chemical-reaction theory of Taggart and his coworkers, however, has lacked direct evidence of the presence of base-metal xanthates at the surface of treated sulfides. A study of these coatings has been in progress at the Montana School of Mines for the past several years. Although the study is still far from complete, the results obtained appear of sufficient interest to be presented at this time.

The study of the coatings formed at the surface of sulfide minerals has been based largely on the examination and analysis of substances leached from the mineral by suitable organic solvents. The choice of a solvent for a given type of coating has not been difficult, since it has been possible to prepare the coating material in bulk and to study its solubility characteristics before embarking on mineral-leaching experiments, the only difficulty being that no suitable solvents were found for some substances, cuprous ethyl xanthate for instance. However, many new substances of unknown composition were obtained which made things more difficult. Furthermore, the quantity of coating substance to be leached away has often been so small as to make analysis difficult if not unreliable. Again, the chemistry of the substances involved—that is, their decomposition, association, solubilities, etc.—is almost unknown, and has had to be built up from the ground, stone by stone. Much work remains to be done in this field. As an instance, it might be mentioned that there is many years' work for a good-sized staff in the precise determination of the solubilities of xanthates in the limited assortment of solvents available in a small laboratory.

EXPERIMENTS WITH GALENA

Some experiments dealing with the reaction of galena with amyl and ethyl xanthates have already been reported,(13) showing that the base-metal xanthate was extracted from the mineral if extraction took place shortly after the reaction, but that sulfur and oils were extracted if extraction was delayed either by adding the reagent previous to the grinding operation or by allowing some time to lapse after addition of the reagents. Although the oil obtained was not definitely identified, it was thought to be at least in part the dixanthogen (alkyl thioformate disulfide) corresponding to the xanthate used. Accordingly experiments with galena were made both with a xanthate (potassium n-amyl xanthate) and the corresponding dixanthogen.(7b) The mineral after cleaning by the Wilkinson method (13b) was ground in bacilli jars with the reagent for various lengths of time. After grinding the aqueous solution was separated from the ground mineral by filtration and analyzed for xanthate(7c) and for other anions by the method of Knoll;(12) at the same time the mineral was leached in the cold successively with acetone, benzene and carbon disulfide for extraction of reaction products. These products were separated by differences in solubility or in ability to form compounds with other ingredients added independently. Whenever sufficient quantities of extracted products were available analyses were made for lead and sulfur. Lead analyses were made by the usual volumetric method; sulfur analyses were made gravimetrically after burning the sample in a Parr-Burgess bomb. In some instances elemental sulfur in oils containing also organically bonded sulfur was determined by shaking a benzene solution of the oil with mercury, an operation resulting in the formation of mercuric sulfide apparently from elemental sulfur only (ferric xanthate, however, constitutes an exception, perhaps because of its ready decomposition(18e)). The elaborate details of procedure are presented elsewhere.(7)

Two series of tests were conducted, one using n-amyl dixanthogen as the reagent and the other using potassium n-amyl xanthate. The same amount of reagent was used in each test but the time of grinding was varied between 2½ and almost 100 hr. The results obtained are presented in Tables 2 and 3.

Table 2 shows that the following substances are extracted from galena ground with potassium amyl xanthate: lead xanthate, sulfur and an oil. The oil, obviously, is not a pure substance, but is composed of at least two constituents, as shown by the decrease in its refractive index from 1.541 to 1.523 with increase in time of grinding. The amount of lead xanthate extracted increases with time of grinding up to almost one gram (obtained in 10¾ hr.) then decreases to the vanishing point. The amounts of sulfur and oil increase consistently with time, the amount of sulfur more than that of oil. Also, the index of refraction of the oil decreases with time of grinding, showing that the proportions of its constituents are changing. It is possible that some oily compound high in bonded sulfur is decomposing to yield elemental sulfur and an oily compound having a lower content of sulfur.

TABLE 2.—*Effect of Time of Grind on Products of Reaction between Potassium n-amyl Xanthate (2.480 Grams) and Galena (75 Grams) Ground in a Bacilli-type Porcelain Jar*

Time of Grind, Hr.	Potassium Amyl Xanthate Abstracted, Grams	Lead Xanthate, Grams	Brown Residue, Grams	Sulfur, Grams	Oil, Grams	Refractive Index of Oil
2½	0.29	0.214	0.000	0.000	0.030	
6	0.82	0.477	0.000	0.030	0.050	
10¾	1.47	0.980	0.000	0.076	0.089	1.541
21	1.98	0.891	tr.[a]	0.084	0.082	1.536
40	2.08	0.169	0.000	0.181	0.182	1.532
92	2.26	0.079	0.000	0.320	0.224	1.523

[a] Less than 0.001 grams.

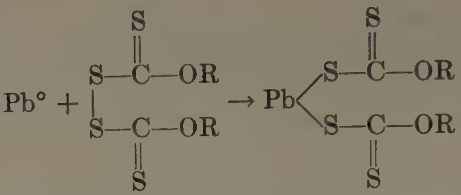
TABLE 3.—*Effect of Time of Grind on Products of Reaction between n-amyl Dixanthogen (2.000 Grams) and Galena (75 Grams) Ground in a Bacilli-type Porcelain Jar*

Time of Grind, Hr	Residual Oil, Grams	Lead Xanthate, Grams	Red-brown Residue, Grams	Decomposition Oil, Grams	Elemental Sulfur, Grams	Refractive Index of Oil
2½	1.0662	0.482	0.005		0.000	1.580
5	0.1646	1.864	0.031		0.000	1.567
10½		2.010	0.052	0.132	0.000	1.542
15		1.581	0.187	0.203	0.020	1.538
20		0.588	0.000	0.426	0.153	1.510
40		0.000	0.000	0.502	0.300	1.507
94		0.000	0.000	0.873	0.480	1.490

Results similar to those obtained with amyl xanthate are obtained with amyl dixanthogen, as may be seen from Table 3. The only difference is provided by the red-brown substance extracted from dixanthogen-treated galena. The large amount of lead xanthate extracted from the treated mineral is difficult to explain. Four interpretations may be considered:

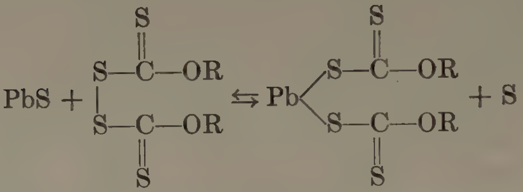
1. The dixanthogen is reduced in some problematical way to xanthate, and the xanthate reacts metathetically with the oxidation coatings at the surface of the galena.

2. The dixanthogen reacts directly with uncharged lead atoms at the surface of the mineral:



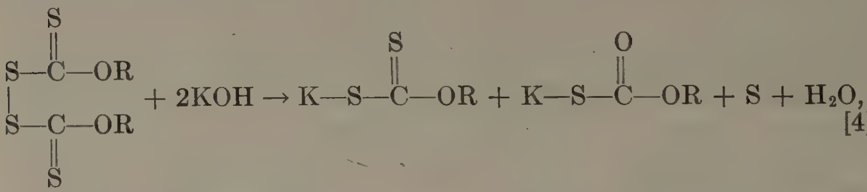
$$\mathrm{Pb}^{\circ} + \begin{array}{l} \mathrm{S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ | \\ \mathrm{S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} \rightarrow \mathrm{Pb}\begin{array}{l} \diagup \mathrm{S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ \diagdown \mathrm{S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} \qquad [2]$$

3. The dixanthogen reacts with lead sulfide to yield lead xanthate and sulfur:



$$\mathrm{PbS} + \begin{array}{l} \mathrm{S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ | \\ \mathrm{S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} \leftrightarrows \mathrm{Pb}\begin{array}{l} \diagup \mathrm{S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ \diagdown \mathrm{S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} + \mathrm{S} \qquad [3]$$

4. The dixanthogen is decomposed by alkali to xanthate, monothiocarbonate and sulfur,(15)



$$\begin{array}{l} \mathrm{S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ | \\ \mathrm{S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} + 2\mathrm{KOH} \rightarrow \mathrm{K{-}S{-}\overset{\overset{S}{\|}}{C}{-}OR} + \mathrm{K{-}S{-}\overset{\overset{O}{\|}}{C}{-}OR} + \mathrm{S} + \mathrm{H_2O}, \qquad [4]$$

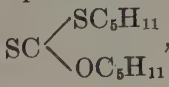
the xanthate and monothiocarbonate subsequently reacting with the mineral metathetically, and the reaction product being a mixture of lead xanthate and lead monothiocarbonate.

None of these interpretations is acceptable at present because there is evidence against each while direct evidence in favor is still lacking. The absence of anions (sulfate, hydroxyl, carbonate or reducing) in the aqueous filtrate from the treated mineral argues against the first and the fourth interpretations, especially if it be remembered that substantial concentrations of sulfate, hydroxyl, carbonate and reducing ions are obtained if potassium xanthate is used in place of dixanthogen (Table 4). There is no evidence whatever for the presence of uncharged lead atoms at the surface of galena; this argues against the second explanation. The absence of sulfur in the leach liquors from galena treated with dixanthogen for short periods of time, from which much lead xanthate was extracted, and the presence of considerable sulfur in the leach liquors from galena treated with dixanthogen for long periods of time from which no lead xanthate was extracted, indicates that the sulfur in the leach liquors has another genesis than a production parallel to that of lead xanthate; this argues against the third and the fourth proposed interpretations. One must therefore consider the problem of the reaction of dixanthogen with galena as being still much of a puzzle.

TABLE 4.—*Analysis of Filtrate from Reaction between Potassium n-amyl Xanthate* (2.48 *Grams or* 12.3 *Milliequivalents*) *and Galena* (75 *Grams*) *Ground for Various Lengths of Time*
ALL IONS STATED AS MILLIEQUIVALENTS

Time of Grind, Hr.	pH	Total Reducing Ions	Xanthate Ion	Reducing Ions Other than Xanthate	Sulfate Ions	Hydroxyl Ions	Carbonate Ions	Total Ions
2½	11.0	11.0	10.8	0.2	0.6	0.9	1.1	13.6
6	11.0	8.6	8.2	0.4	0.1	0.3	2.3	11.3
10¾	11.6	6.0	5.0	1.0	0.4	0.7	3.5	10.6
21	11.2	4.0	2.5	1.5	0.6	0.5	4.0	9.1
40	11.0	3.9	2.0	1.9	0.4	0.2	4.2	8.9
92	11.0	3.2	1.2	2.0	0.8	0.3	4.6	8.9

The formation of the red-brown substance appearing in Table 3 as one of the leach products from galena treated with dixanthogen is perhaps significant in this connection. This substance was not observed (except in one instance, as a "trace") in the tests in which potassium xanthate was used in place of dixanthogen. This substance, although apparently homogeneous when in solution in benzene, breaks down on drying (perhaps accompanied by oxidation), yielding lead xanthate, sulfur and lead sulfide. By itself this substance might well be overlooked, but its resemblance to numerous substances extracted from chalcocite treated with xanthate or dixanthogen lends some weight to the belief that it represents an intermediate compound of a lead polysulfide with lead xanthate, or that it is perhaps simply lead disulfide mixed with lead xanthate. The appearance of the substance agrees with Duncan and Ott's description(14) of the appearance of lead disulfide; however, the unknown red-brown substance is soluble in benzene whereas lead disulfide was prepared by Duncan and Ott by precipitation from benzene.

The lead xanthate formed on galena is decomposed in time, yielding sulfur and an oil termed here "decomposition oil" in contrast with residual dixanthogen ("residual oil"). The amount of sulfur produced increases to slightly more than one-half of the sulfur in the dixanthogen. The discrepancy from an exact equivalence to one-half the sulfur in the dixanthogen used might be ascribed to experimental error. The "residual oil" separated from sulfur by repeated solution in a mixture of 90 per cent methanol and 10 per cent water and evaporation contained 29.6 per cent and 28.5 per cent sulfur in two different experiments. This sulfur may be compared with the sulfur contained in the amyl ester of amyl xanthic acid,  $SC\left\langle\begin{matrix}SC_5H_{11}\\OC_5H_{11}\end{matrix}\right.$, 27.4 per cent.

The tests show that lead amyl xanthate forms at the surface of galena when either potassium xanthate or dixanthogen is added. They show also that while the lead xanthate from potassium xanthate forms by metathesis, that from dixanthogen has probably a different genesis. The tests show, also, that the lead amyl xanthate decomposes in a few hours, even in an atmosphere containing but a limited amount of oxygen to yield sulfur and an oil containing sulfur. Sulfur, the alkyl ester of the alkyl xanthic acid and lead carbonate, could originate from lead xanthate by oxidation of the latter with oxygen, but the quantitative data available do not at present justify the writing of a reaction. The tests were conducted in circuits almost strictly neutral (pH 6.8 ± 0.1*) whenever dixanthogen was used, and significantly alkaline (pH about 11.0*) whenever xanthate was used, so that the results may be thought to typify what happens in the usual range of galena-floating operations performed in practice, using amyl compounds as collectors. Since, furthermore, the earlier experiments of Wilkinson(13b) also indicated that sulfur and oils are end products in the treatment of galena with ethyl xanthate, the later results may be taken to have general significance.

Experiments with Chalcocite

Experiments of the same general nature as those just described for galena were conducted with chalcocite. The mineral was first concentrated by jigging and picking with forceps, crushed, tabled, deslimed and leached with acid and ammonia. The mineral so obtained was practically free of any impurity other than cupric sulfide (largely in solid solution) and interstitial copper carbonate not reached by the lixiviants. It was used as such in some tests, and also after a final purification consisting of melting in a crucible under a reducing atmosphere. This last operation resulted in the formation of additional Cu_2S from CuS and CuO (derived from the copper carbonates) and also possibly by direct elimination of sulfur from CuS. The final product contained much less cupric copper and a very small quantity of iron almost certainly in solution in the cuprous sulfide. The acid and ammonia-cleaned mineral on the one hand and the melted product on the other behaved much in the same manner.(16a)

The experimentation was largely conducted with n-amyl compounds since copper amyl xanthate is soluble in some organic solvents (though to a limited extent) whereas the ethyl compound is practically insoluble in all organic solvents. Experiments were also made with the n-octyl compound whose copper derivatives appear more soluble in organic liquids than the amyl derivatives themselves.

Several different substances were extracted from the treated mineral. One of them was the lemon-yellow cuprous xanthate, and there were also two red substances, one orange-brown and two brown-black, all five containing copper and sulfur. These various products were separated on the basis of difference in solubility in organic solvents. All appeared to be amorphous. However, their composition appeared sufficiently definite from test to test to lead the writers to believe that they are definite compounds and not merely mixtures. All analyses(16b) were made for both copper and sulfur after oxidation in a Parr-Burgess bomb by the usual electrolytic and gravimetric methods, respectively; attempts at determining hydrogen and carbon by the combustion-train method failed because of the low-temperature decomposition of the unknowns, yielding volatile products before the apparatus was hot enough to insure complete combustion. The combustions did show positively the presence of carbon and hydrogen in the compounds; alkyl sulfides were produced and identified by their characteristic odor in the products of the incomplete combustion.

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* Determined colorimetrically with LaMotte indicators.

No elemental sulfur was obtained from any of the chalcocite-leaching operations and no dixanthogen or any other oil was extracted in substantial amount.

TABLE 5.—*Solubility of Compounds Extracted from Chalcocite Treated with Xanthates or Dixanthogens*

Compound	Amyl No. 1	Amyl No. 2	Amyl No. 3	Amyl No. 4	Amyl No. 5	Amyl No. 6	Octyl No. 6	Synthetic	
								Cuprous n-amyl Xanthate	Cuprous n-amyl Trithiocarbonate
Color	Bright Yellow	Orange Brown	Red	Red	Dark Brown	Brown Black	Brown Black	Bright Yellow	Red
Solubility in water	F	F	F	F	F	F	F	F	F
95 per cent ethanol	E	E	E	E	F	F	F	E	E
Absolute methanol	E	E	D	D	D	D	F	E	E
Acetone	D	D	B	B	B	E	F	D[a]	E
Diethyl ether	D	D	D	B	B	B	C	D[b]	D
Carbon disulfide	C	C	B	B	B	B	B	C[c]	B
Benzene	C	C	B	B	B	B	C	C[d]	C
Petroleum ether	F	F	D	B	B	B	B	F[f]	D

A denotes extreme solubility; B, high solubility; C, medium-low solubility; D, poor solubility; E, very slight solubility; F, insolubility (practically speaking).

[a] 16 mg. per liter. [b] 15 mg. per liter. [c] 7.3 grams per liter. [d] 388 mg. per liter. [f] Less than 1 mg. per liter.

Table 5 presents a qualitative list of the solubilities of the various compounds obtained and Table 6 a list of the copper and sulfur content of the compounds.

No certainty could be had that the various products isolated from chalcocite treated with potassium amyl xanthate or amyl dixanthogen were pure, since the method of separating them was dependent upon the relative solubilities of the compounds in acetone, diethyl ether, benzene and petroleum ether. It is surprising, therefore, that a concordance as good as that suggested by Table 6 is obtained between the chemical analyses obtained and those calculated for the compounds $Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$; $Cu_2S.Cu_2[SC(O)OC_5H_{11}]_2$; $2Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$; $3Cu_2S.Cu_2[SC(S)OC_5H_{11}[_2$: and $3Cu_2S.Cu_2[SC(S)OC_8H_{17}]_2$, respectively.

TABLE 6.—*Comparison of Chemical Composition of Compounds Extracted from Chalcocite Treated with Xanthates or Dixanthogens, with Composition of Compounds Postulated to Correspond to Them*

Substance	Composition Obtained by Analysis (on Samples Obtained Separately), Per Cent		Chemical Composition, Calculated, Per Cent	
	Cu	S	Cu	S
Cuprous n-amyl xanthate			28.0	28.2
Amyl No. 1	28.5	30.5		
	28.9			
	27.7			
Amyl No. 2	34.6	29.3		
$Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$			41.5	26.1
Amyl No. 3	37.3	24.7		
Amyl No. 4	42.8	18.7		
	40.9	17.4		
$Cu_2S.Cu_2[SC(O)OC_5H_{11}]_2$			43.7	16.5
Amyl No. 5	48.3			
	47.4	24.1		
	48.3			
	47.5	23.3		
$2Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$			49.5	24.9
Amyl No. 6[a]	52.5			
	51.4			
	51.5	23.0		
	50.3	24.7		
	52.4	24.8		
$3Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$			54.8	24.0
Octyl No. 6	49.6	22.1		
	50.2	22.1		
$3Cu_2S.Cu_2[SC(S)OC_8H_{17}]_2$			50.0	22.1

[a] A careful molecular-weight determination of this product was made by the Cottrell ebullioscopic method using a benzene solution of the product. The molecular weight was found to be 479, or about one-half of the molecular weight (930) required for $3Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$. This may be taken as an indication that the compound dissociates upon heating or that it contains a small bulk of a substance of low molecular weight. Molecular-weight determinations by the freezing-point method were unsatisfactory because of the insufficient solubility and high molecular weight of the substance in freezing benzene.

Still better "fits" by way of formulas can be obtained in some cases, but not in all, by considering some of the copper as cupric; that is by postulating compounds of the type $x\mathrm{CuS}.y\mathrm{Cu}_2X_2$ or $x\mathrm{CuS}.x'\mathrm{Cu}_2\mathrm{S}.y\mathrm{Cu}_2X_2$.

The extraction product amyl No. 2 appears especially impure, containing apparently a large proportion of a yellow constituent, which it was not found possible to separate from the orange-brown substance. This extraction product can be regarded temporarily as a mixture of cuprous xanthate and the substance $Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$.

The extraction product amyl No. 4 approximates fairly closely $Cu_2S.Cu_2[SC(O)OC_5H_{11}]_2$, which appears to be derived from an amyl monothiocarbonate rather than from an amyl xanthate (or dithiocarbonate).

The extraction product amyl No. 3, which resembles No. 4 considerably from a solubility standpoint, may be either a mixture of $Cu_2S.Cu_2[SC(O)OC_5H_{11}]_2$ with cuprous amyl xanthate or a mixture of $Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$ with cuprous amyl monothiocarbonate, or essentially a compound derived partly from a xanthate and partly from a monothiocarbonate, having a formula of the type $Cu_2S.Cu_2[SC(O)OC_5H_{11}][SC(S)OC_5H_{11}]$. It is noteworthy that the solubility relations of amyl No. 3 and amyl No. 4 indicate that they are the most polar of the various extraction products. The products amyl No. 3 and amyl No. 4 are also relatively unstable, their appearance changing somewhat as a result of solution and evaporation of the solution. This fact together with their higher polarity is in harmony with their being derived from a thiocarbonate containing but one sulfur atom per carbonate group instead of two.

The extraction products amyl No. 5 and amyl No. 6 approximate closely the formulas $2Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$ and $3Cu_2S.Cu_2[SC(S)OC_5H_{11}]_2$.

Likewise the extraction product octyl No. 6, which is in every respect similar to amyl No. 6, has a composition corresponding very closely to $3Cu_2S.Cu_2[SC(S)OC_8H_{17}]_2$.

Table 7 and Table 8 present a summary of the reaction products isolated from chalcocite treated with n-amyl potassium xanthate and dixanthogen for various lengths of time upon extraction of the treated mineral with organic solvents. These tables show that there is a gradual progression in the character of the product extracted from cuprous amyl xanthate to amyl No. 6, and, indeed, possibly to some compound higher in Cu_2S content than amyl No. 6, which it has not been possible to extract by leaching.

Compounds of the types of amyl Nos. 2, 3, 4, 5 and 6 and of octyl No. 6 can be interpreted as forming either through the association of Cu_2S with Cu_2X_2 or through some decomposition of Cu_2X_2.

Association Hypothesis.—It may be postulated that Cu_2S on contact with Cu_2X_2 associates with the latter to give various $Cu_2S.Cu_2X_2$ compounds. But if that is true, it is difficult to understand why such an association must wait, in order to proceed, until disappearance of the xanthate ion from the solution in which the mineral is immersed—and yet such are the facts.

Table 7.—*Effect of Time of Grinding on Products Extracted from Chalcocite (50 Grams) Treated with* 0.01 *Mol* (2.02 *Grams*) *of Potassium n-amyl Xanthate*

Substance	Time of Grinding, Hr.							
	1¼	2½	5	10	20	30	40	96[a]
	Product Extracted, Grams							
Amyl No. 1 and 2	1.670	1.524	1.224	0.405	0.200	0.095	0.002	0.000
Amyl No. 3, 4 and 5	0.000	0.171	0.246	0.793	0.303	0.060	0.000	0.000
Amyl No. 6	0.000	0.000	0.177	0.519	0.650	0.402	0.020	0.000
Oil	trace	trace	trace	trace	0.000	0.000	0.000	0.000
Tar	0.000	0.000	0.000	0.000	0.000	0.000	0.000	trace
Sulfur	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

[a] Mineral still highly floatable.

Table 8.—*Effect of Time of Grinding on Products Extracted from Chalcocite (50 Grams) Treated with* 0.005 *Mol* (1.63 *Grams*) *of n-amyl Dixanthogen*

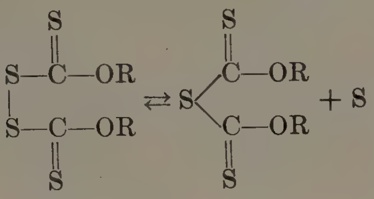
Substance	Time of Grinding, Hr.		
	5	20	96[a]
	Product Extracted, Grams		
Amyl No. 1	1.95	0.75	0.000
Amyl No. 3 and No. 4	0.000	0.000	0.000
Amyl No. 5 and No. 6	0.150	0.350	0.000
Oil	0.040	0.004	0.000
Sulfur	0.000	0.000	0.000

[a] Mineral highly floatable.

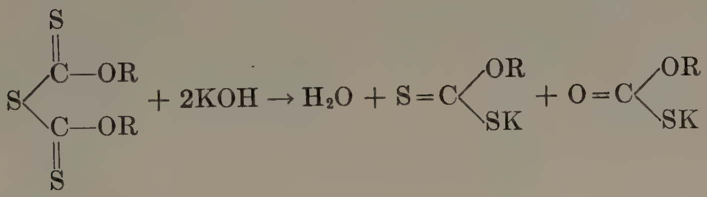
Furthermore, it may be argued that there is not a shred of evidence to show that Cu_2S does associate with organic copper salts. On the other hand it may, of course, be pointed out that copper xanthate precipitated in alcohol solution from potassium xanthate by cupric chloride dissolves in excess cupric chloride, first yielding an orange-red coating on the precipitated cuprous xanthate and later a yellow-green solution of some complex salt; by analogy complexes of Cu_2S with Cu_2X_2 are not unexpectable.

Decomposition Hypothesis Involving Alkali Alone.—Two different decomposition hypotheses can be advanced, one of which postulates decomposition by the alkali in solution, and the other by the alkali in solution in the presence of oxygen.

In connection with the decomposition hypothesis involving alkali alone, it may not be amiss to recall that dixanthogen in acetone solution decomposes in the presence of alkali (eq. 4). This reaction may be regarded as occurring in two steps:



[5a]



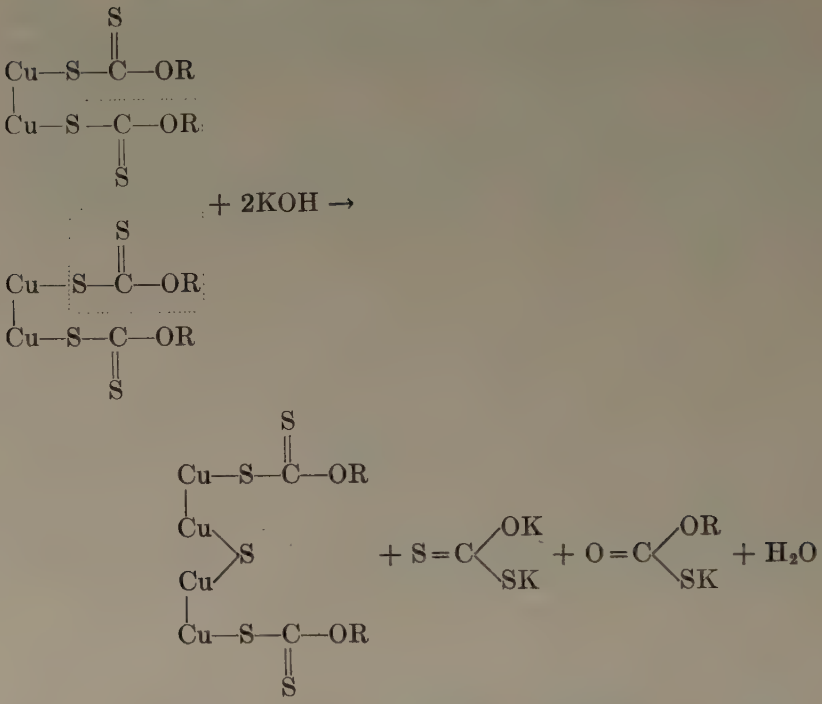
[5b]

In the tests with chalcocite in which xanthate was used a pH of about 11 was naturally obtained, indicating a substantial concentration of hydroxyl ions. In tests involving either xanthate or dixanthogen, the process of decomposition of the cuprous amyl xanthate was speeded up by the addition of alkali, as may be seen from Table 9.

TABLE 9.—*Effect of Alkali on Character of Substance Extracted from Chalcocite* (50 *Grams*) *Treated with* 0.01 *Mol Potassium n-amyl Xanthate or Equivalent n-amyl Dixanthogen during Grinding Period of* 20 *Hours*

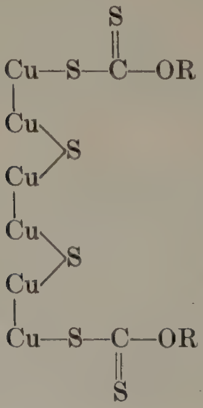
Reagent	Xanthate	Xanthate	Dixanthogen	Dixanthogen
Alkali added, mol	0.000	0.100	0.000	0.010
Products extracted:				
Cuprous xanthate	0.200	0.000	0.750	0.310
Amyl No. 3 and 4	0.024	0.000	0.000	0.016
Amyl No. 5	0.278	0.000	0.070	0.359
Amyl No. 6	0.650	0.000	0.280	0.569
Tar	0.000	0.020	0.000	0.000

From these facts it is possible to postulate with fair likelihood of being correct that after an initial formation of cuprous xanthate, that substance is decomposed by alkali as follows:

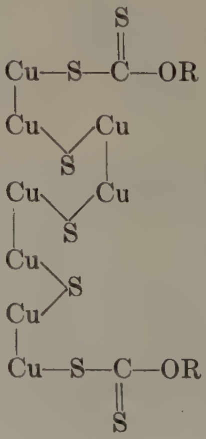


[6]

This decomposition consists, of course, in splitting off a "monoxanthogen" (alkyl thioformate monosulfide) group from two contiguous cuprous xanthate molecules, then in decomposing this monoxanthogen molecule with alkali (as in eq. 5*b*) into one xanthate molecule and one monothiocarbonate molecule. The same reaction may take place, of course, between cuprous xanthate and the main product of reaction 6, to yield:



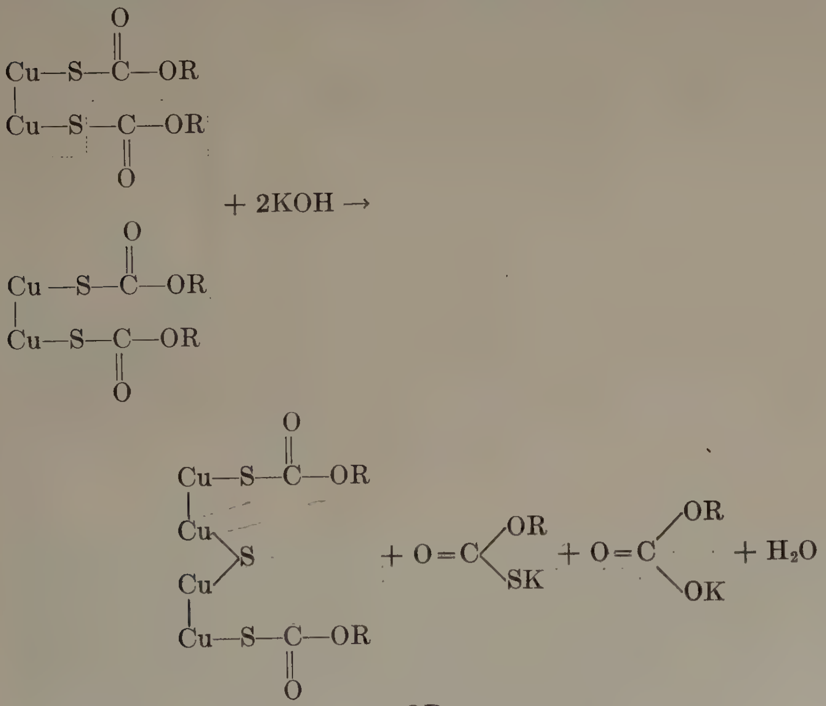
or



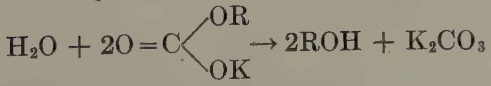
which are thought to be typified by the substances amyl No. 5 and amyl No. 6. In fact, similar reactions may yield products with a Cu_2S content higher than $3Cu_2S.Cu_2[SC(S)OR]_2$, although it would appear that the generating reaction should be increasingly difficult of accomplishment with increasing complexity in the reacting molecules.

But the xanthate ion and the monothiocarbonate ion produced by reaction 6 and by those yielding the compounds 5 and 6 become reinvested, as it were, yielding cuprous xanthate and cuprous monothiocarbonate by metathesis with further oxidation layers produced on the chalcocite.(10) This means that the eventual disintegration of the cuprous xanthate at the surface of the chalcocite is retarded by continuous regeneration of a part of the coating.

Monothiocarbonates are much less stable than xanthates. Reaction 7 may therefore be considered as more likely than reaction 6, from which it differs only in that monothiocarbonate and carbonate replace dithiocarbonate (xanthate) and monothiocarbonate. The alkyl carbonate, of course, is unstable and hydrolyses, yielding alcohol and alkali carbonate:



$$\begin{array}{l}\mathrm{Cu{-}S{-}C({=}O){-}OR}\\ \;|\\ \mathrm{Cu{-}S{-}C({=}O){-}OR}\\[4pt] \mathrm{Cu{-}S{-}C({=}O){-}OR}\\ \;|\\ \mathrm{Cu{-}S{-}C({=}O){-}OR}\end{array} + 2\mathrm{KOH} \rightarrow \begin{array}{l}\mathrm{Cu{-}S{-}C({=}O){-}OR}\\ \;|\\ \mathrm{Cu}\diagdown\\ \quad\;\; \mathrm{S}\\ \mathrm{Cu}\diagup\\ \;|\\ \mathrm{Cu{-}S{-}C({=}O){-}OR}\end{array} + \mathrm{O{=}C}\begin{array}{l}\diagup\mathrm{OR}\\ \diagdown\mathrm{SK}\end{array} + \mathrm{O{=}C}\begin{array}{l}\diagup\mathrm{OR}\\ \diagdown\mathrm{OK}\end{array} + \mathrm{H_2O} \quad [7]$$

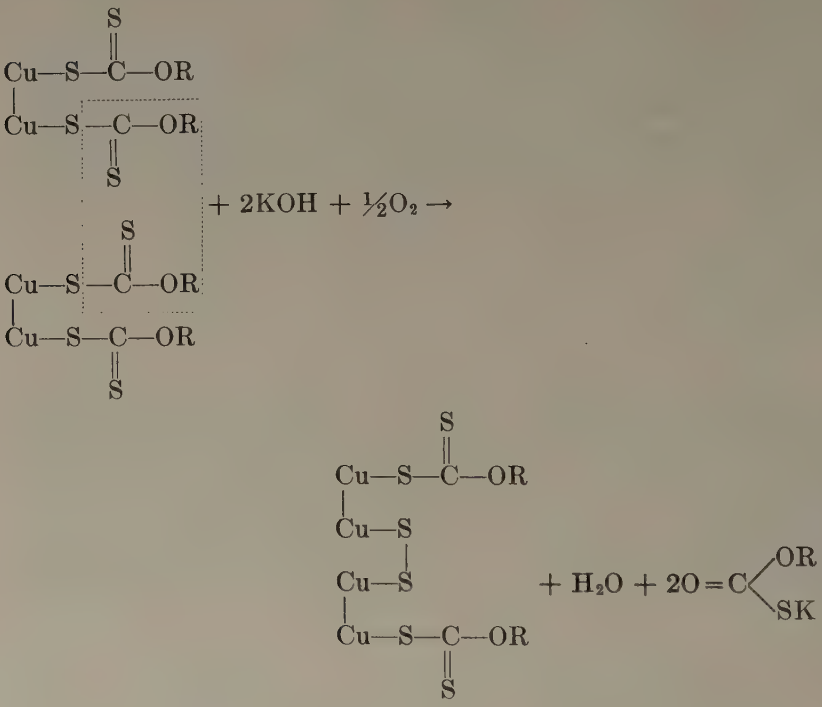


$$\mathrm{H_2O} + 2\mathrm{O{=}C}\begin{array}{l}\diagup\mathrm{OR}\\ \diagdown\mathrm{OK}\end{array} \rightarrow 2\mathrm{ROH} + \mathrm{K_2CO_3} \quad [8]$$

A compound such as $Cu_2S.Cu_2[SC(O)OR]_2$ would correspond to the intermediate, relatively unstable red compound extracted from the chalcocite (amyl No. 4). It might be noted at this point that equations 7 and 8 indicate that carbonate ion and alcohol should be obtained from the mineral. Both of these products have been identified.(16)

Complex compounds derived partly from cuprous xanthate and partly from cuprous thiocarbonate can perhaps be expected to form.

Decomposition Hypothesis Involving Alkali and Oxygen.—It is possible that the decomposition of cuprous xanthate on chalcocite involves oxygen besides hydroxyl ion. If that is the case one might expect the decomposition product to contain cupric copper with cuprous copper, instead of cuprous copper only:



$$\begin{array}{l} \mathrm{Cu{-}S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ | \\ \mathrm{Cu{-}S{-}\underset{\underset{S}{\|}}{C}{-}OR} \\ \\ \mathrm{Cu{-}S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ | \\ \mathrm{Cu{-}S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} + 2KOH + \tfrac{1}{2}O_2 \rightarrow \begin{array}{l} \mathrm{Cu{-}S{-}\overset{\overset{S}{\|}}{C}{-}OR} \\ | \\ \mathrm{Cu{-}S} \\ \quad\;\; | \\ \mathrm{Cu{-}S} \\ | \\ \mathrm{Cu{-}S{-}\underset{\underset{S}{\|}}{C}{-}OR} \end{array} + H_2O + 2O{=}C\begin{array}{l} \diagup OR \\ \diagdown SK \end{array} \qquad [9]$$

Evidence that oxygen plays a part in the decomposition of cuprous xanthate at the surface of chalcocite is afforded by two parallel tests, in one of which grinding was conducted in the presence of the usual limited volume of air at atmospheric pressure (0.022 mols O_2 having an oxidizing power of 0.088 equivalent electrons) and the other in the presence of very much less oxygen (0.00048 mols O_2 having an oxidizing power of 0.0019 equivalent electrons). The results obtained are presented in Table 10.

If such a reaction as postulated by eq. 9 can be proved, the formulas designed to fit the new substances whose isolation is reported in this paper would have to be modified to make part of the copper cupric rather than all cuprous. (See Table 6.)

It is the opinion of the writers that the decomposition hypothesis involving oxygen as a factor is as yet insufficiently proved to be considered a theory, but that some type of "decomposition" hypothesis appears necessary.

TABLE 10.—*Effect of Oxygen on Character of Substance Extracted from Chalcocite* (50 *Grams*) *Treated with* 10 *Millimols Potassivm n-amyl Xanthate during Grinding Period of* 20 *Hours in Porcelain Grinding Mill of* 500-*gram Capacity*

Atmosphere	Air[a]	Vacuo[b]
Odor	Amyl alcohol	Xanthate
Residual agent, millimols	0.0	6.7
Ions in solution, milliequivalents		
Hydroxyl	2.05	0.27
Carbonate	3.38	1.05
Sulfate	0.94	0.18
Reducing ions other than xanthate	0.10	0.00
Products extracted from mineral, grams:		
Cu_2X_2	0.40	0.25
Amyl No. 3 and 4	0.10	0.01
Amyl No. 5 and 6	0.70	0.05

[a] Containing 0.022 mols O_2.

[b] Containing 0.00048 mols O_2.

The writers do not regard the chain formulas appearing on pages 334 to 336 as established. Indeed, the great solubility of some of the newly described compounds in nonpolar liquids such as petroleum ether and the insolubility in those liquids of either copper sulfides or cuprous xanthates suggest a structure for the unknown substances such that the copper and sulfur atoms are "buried" in the central part of the molecule so as to fail to exert a marked effect on the molecule as a whole in respect to the solvent. Ring structures of copper and sulfur atoms, for example, are not necessarily to be considered as ridiculous, any more than structures featuring coordination valencies.

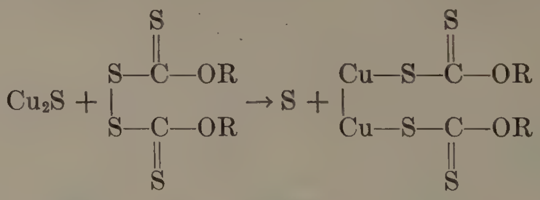
It is interesting, in connection with the hypothesis of cuprous xanthate decomposition advanced here, that cuprous xanthate in bulk ground with aplite for 20 hr. is recovered without decomposition.(16) This can perhaps be ascribed to the fact that practically all the cuprous xanthate molecules are available for reaction or decomposition when spread as a thin layer at the surface of chalcocite, whereas but a small fraction is so available in bulk cuprous xanthate; also, the substrate itself may in some unknown manner affect the ability of the coating to decompose.

Considering now the initial reaction between the collector and the mineral, it is interesting to note the practical absence of ions in solution where dixanthogen has been used, but the presence of a considerable concentration of ions where xanthate has been used (Table 11). This may be taken as indicating that in neutral circuits dixanthogen reacts directly with chalcocite with the formation of sulfur and cuprous xanthate

TABLE 11.—*Ions in Liquor from Grinding Chalcocite* (50 *Grams*) *with* 10 *Millimols* (2.02 *Grams*) *Potassium n-amyl Xanthate or Equivalent* (1.63 *Grams*) *Dixanthogen*

ALL IONS EXPRESSED IN MILLIEQUIVALENTS

Ions	Xanthate	Dixanthogen
pH	11.4	7.3
Hydroxyl	0.75	0.00
Carbonate	4.59	0.08
Sulfate	1.48	0.27
Reducing anions	0.02	0.00
Total anions	6.74	0.35
Potassium	6.76	Not determined



$$\mathrm{Cu_2S} + \begin{array}{l} S \\ \| \\ S-C-OR \\ | \\ S-C-OR \\ \| \\ S \end{array} \rightarrow S + \begin{array}{l} S \\ \| \\ Cu-S-C-OR \\ | \\ Cu-S-C-OR \\ \| \\ S \end{array} \qquad [10]$$

In the experiments under discussion, the dixanthogen was present during the grinding operation, no doubt as a fine emulsion in water. Minute droplets of the agent therefore had a splendid opportunity to adhere to the newly opened sulfide surface as soon as that surface became available, either by just bumping against it or by spreading from the outside of the particle in process of fragmentation, just as oleic acid spreads at the surface of water. Since cuprous sulfide may contain in solution a substantial amount of sulfur in excess of that required for cuprous sulfide,(17) equation 10 does not necessarily postulate the creation of another solid phase (S or CuS) besides the cuprous xanthate phase. The sulfur atoms may simply diffuse within the Cu_2S lattice as a solid solution beyond the reach of solvents. Further evidence for this equation is found in the fact that dixanthogen fails to react with malachite,(5b) that dixanthogen is extracted from malachite treated with alkali xanthates, even in alkaline pulps under conditions otherwise promoting the decomposition of dixanthogen,(16e) but that dixanthogen is not extracted in significant amount from chalcocite treated with either xanthate or dixanthogen in neutral or in alkaline pulps.

Fig. 1 shows what ions are found in the liquor from the treatment of chalcocite with n-amyl xanthate for increasing lengths of time. This figure brings out the following facts:

1. The total anion concentration is too low to equal that of the anion added in the system. This can be explained by assuming, without evidence to be sure, that there is ion adsorption by some constituent of the system.

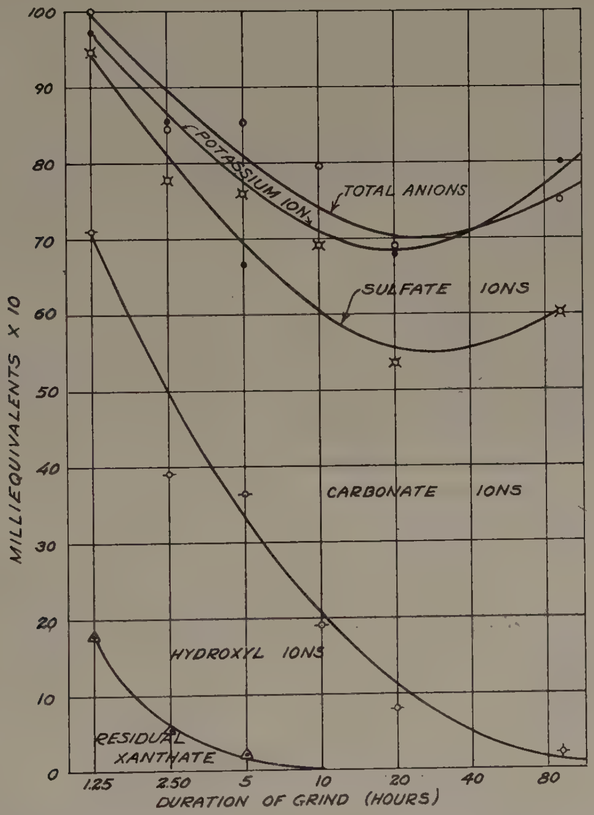


FIG. 1.—IONS IN SOLUTION CHALCOCITE +0.01 MOL K-N-AMYL XANTHATE.

2. The total ion concentration decreases as grinding continues up to a certain point, then increases again.

3. The residual xanthate-ion concentration is small even with short grinds.

4. The hydroxyl-ion concentration decreases with grinding, as if hydroxyl ion were consumed in a reaction.

5. The carbonate-ion concentration increases, as if continuously generated, even when no further abstraction of xanthate ion is taking place.

Decrease in hydroxyl ion and increase in carbonate ion are both called for by reactions 6, 8 and 9. Since the chalcocite used in the tests from which Fig. 1 was drawn was "furnace chalcocite," and since grinding proceeded in a closed mill, it does not appear possible to account for the considerable carbonate ion in any other way than by the decomposition of cuprous monothiocarbonate (reaction 7). Furthermore, considerable amyl alcohol was observed to have formed upon prolonged grinding of chalcocite treated with amyl xanthate or amyl dixanthogen (evidenced by the odor of the aqueous filtrate, by the surface tension and the frothing qualities of that solution and by the odor of the mineral).

To summarize, it would appear that the mechanism of xanthate action on chalcocite is as follows:

1. The xanthate ion changes place with hydroxyl, carbonate and sulfate ion at the surface of the mineral, yielding thereon cuprous xanthate and dixanthogen.
2. The dixanthogen at the mineral surface reacts with the mineral to produce more cuprous xanthate.
3. Cuprous xanthate reacts with hydroxyl ion in solution with or without oxygen taking part in the reaction to produce intermediate coatings of the type $x\mathrm{Cu_2S}.y\mathrm{Cu_2[SC(S)OR]_2}$ or $x'\mathrm{CuS}.y\mathrm{Cu_2[SC(S)OR]_2}$ together with xanthate and thiocarbonate ions in solution.
4. Cuprous xanthate and cuprous thiocarbonate form by reaction between further oxidation coatings at the surface of the mineral and the xanthate and thiocarbonate ions in solution.
5. Cuprous thiocarbonate reacts with hydroxyl ion in solution with or without oxygen taking part in the reaction, to produce intermediate coatings of the type $x\mathrm{Cu_2S}.y\mathrm{Cu_2[SC(O)OR]_2}$ or $x'\mathrm{CuS}.y\mathrm{Cu_2[SC(O)OR]_2}$ together with thiocarbonate ions, carbonate ions and alcohol in solution.
6. Among the end products of the reaction are carbonate ion and alcohol in solution.

EXPERIMENTS WITH COVELLITE

A few preliminary experiments were conducted(16) with a very pure covellite available only in small quantity. The mineral contained 65.9 per cent Cu, 0.2 per cent insol. and 0.0 per cent Fe, as compared with 66.5 per cent Cu in chemically pure CuS. These experiments showed that from mineral treated with n-amyl xanthate there were extracted cuprous xanthate, dixanthogen and sulfur, together with a small quantity of a dark green amorphous solid possibly related to the brown compounds extracted from xanthate-treated chalcocite. The formation of dixanthogen and of sulfur is significant, in that these two substances are not obtained when chalcocite is used instead of covellite. The presence of sulfur shows that in this respect at least covellite behaves more like galena and pyrite than like chalcocite. Comparative results for malachite, covellite and chalcocite are presented in Table 12.

Table 12.—*Effect of Grinding* 10 *Millimols* (2.02 *Grams*) *Potassium n-amyl Xanthate or Equivalent Dixanthogen* (1.63 *Grams*) *with Chalcocite, Covellite and Malachite*

Ion Content of Filtrates Expressed as Milliequivalents

Mineral	Malachite	Covellite	Covellite	Chalcocite	Chalcocite
Weight of mineral, grams	50	40	40	50	50
Time of grind, hours	20	10	10	10	20
Reagent	Xanthate	Dixanthogen	Xanthate	Xanthate	Xanthate
Odor	Clayey	AmOH	AmOH	AmOH	AmOH
pH	11.2	7.6	11.0	11.5	11.4
Anions in filtrate:					
Total content	8.2		6.57	7.95	6.84
Hydroxyl	3.5		0.64	1.78	0.75
Carbonate	4.7		3.33	5.03	4.59
Sulfate	0.0		0.42	1.02	1.48
Xanthate	0.0		0.00	0.00	0.00
Reducing anions other than xanthate	0.0		2.18	0.12	0.02
Leach products, grams:					
Sulfur	0.000	0.060	0.050	0.000	0.000
Dixanthogen and green substances dispersed in it	0.800	0.641	0.250	0.000	0.000
Cuprous xanthate and orange-brown substance associated with it (Amyl Nos. 1, 2)	1.015	0.650	0.417	0.405	0.200
Amyl Nos. 3, 4, 5	0.000	0.000	0.000	0.793	0.303
Amyl No. 6	0.000	0.000	0.000	0.519	0.650

The difference in the behavior of covellite and chalcocite is perhaps to be ascribed to the facility with which chalcocite acts as an acceptor of sulfur, that element being taken up by mineral whereas it cannot dissolve within covellite.

Although the experiments conducted so far are merely preliminary, it is interesting to note that the results obtained differ from those obtained with chalcocite in such a way as to support the conclusions obtained with that mineral rather than to detract from them.

EXPERIMENTS WITH PYRITE

Early experiments with pyrite(13) showed that pyrite treated with amyl xanthate, or ethyl xanthate, or ethyl dixanthogen either during grinding or after grinding yielded in every case elemental sulfur and oils.

The oils were not determined exactly but were thought to be dixanthogen. The sulfur was equivalent to about one-half the sulfur present in the collector. The reactions involved were believed to be:

1. Pyrite + oxygen + xanthate ion → dixanthogen-coated pyrite + sulfate ion
2. Pyrite + dixanthogen suspension in water → dixanthogen-coated pyrite
3. Dixanthogen-coated pyrite + oxygen → sulfur + miscellaneous volatile compounds.

Since the reactions thus postulated do not appear to involve the production of iron xanthate, a production required by an extension of the Taggart, Taylor and Knoll theory to pyrite, it was thought desirable to allow pyrite to react with an alkali xanthate under conditions favoring as much as possible the production of iron xanthate.

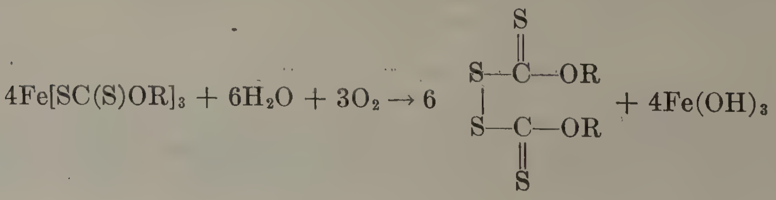
In the first place, attention should be called to the fact that iron xanthate is the ferric compound and not the ferrous compound as is elsewhere stated. An analysis of iron xanthate made from ferrous sulfate and potassium ethyl xanthate gave 13.2 per cent Fe and 45.5 per cent S, against calculated values of 13.3 per cent Fe and 45.9 per cent S for ferric ethyl xanthate.

A preliminary study was made of the chemical properties of ferric xanthates. It was found that these substances are extremely insoluble in water, little soluble in heteropolar organic solvents and very soluble in nonpolar organic solvents.(8a) It was found, furthermore, that ferric xanthates crystallized from benzene or diethyl ether remain practically unchanged in a dessicator, but that they decompose rapidly in a moist atmosphere with the production of ferric hydroxide and xanthic acid:

$$\mathrm{Fe[SC(S)OR]_3 + 3H_2O \rightarrow Fe(OH)_3 + 3HSC(S)OR} \qquad [11]$$

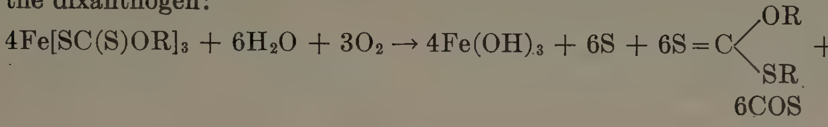
If oxygen is not excluded, the xanthic acid oxidizes readily to dixanthogen. Ferric xanthates dissolved in nonpolar organic solvents keep well except at the boundary between the liquid phase and glass or air where ferric hydroxide precipitates. Moisture appears to be required for this decomposition. In heteropolar organic solvents, such as alcohols or acetone, especially the latter, ferric xanthates decompose rapidly, largely by oxidation.

In acid acetone solutions the hydrolysis is followed or accompanied by oxidation to dixanthogen:



$$\mathrm{4Fe[SC(S)OR]_3 + 6H_2O + 3O_2 \rightarrow 6} \begin{array}{lcl} & \mathrm{S} & \\ & \| & \\ \mathrm{S}- & \mathrm{C} & -\mathrm{OR} \\ | & & \\ \mathrm{S}- & \mathrm{C} & -\mathrm{OR} \\ & \| & \\ & \mathrm{S} & \end{array} + \mathrm{4Fe(OH)_3} \qquad [12]$$

In alkaline acetone solutions the hydrolysis is followed or accompanied by oxidation to dixanthogen, and that in turn is followed by decomposition of dixanthogen with the production of elemental sulfur and (if ethyl compounds are being used) ethyl ethyl-xanthate. It is not known whether the last step involves an oxidation or a mere decomposition. Writing the equation as involving a mere decomposition of the dixanthogen:



$$4\mathrm{Fe[SC(S)OR]_3} + 6\mathrm{H_2O} + 3\mathrm{O_2} \rightarrow 4\mathrm{Fe(OH)_3} + 6\mathrm{S} + 6\mathrm{S{=}C}\begin{array}{l}\diagup \mathrm{OR}\\ \diagdown \mathrm{SR}\end{array} + 6\mathrm{COS} \qquad [13]$$

The breakdown of ferric xanthate may then be regarded as consisting of several steps: first, a hydrolysis to ferric hydroxide and xanthic acid; second, an oxidation of the xanthic acid to dixanthogen; third, a decomposition of the dixanthogen to sulfur and monoxanthogen; and fourth a decomposition of monoxanthogen to the alkyl ester of the alkyl xanthic acid used and carbon oxysulfide. The last step in the reaction may not be a strict decomposition, but rather an oxidation. In acid circuits the chain of decomposition stops with the production of dixanthogen, but in alkaline circuits it proceeds further.

If in flotation operations pyrite is coated monomolecularly, or even polymolecularly by ferric xanthate, the most excellent opportunity is presented for immediate alteration of the coating, since practically all the coating substance is directly in contact with the modifying agents; namely, water and oxygen. It might be recalled, for instance, that cuprous xanthate as a coating on chalcocite does alter whereas cuprous xanthate in bulk remains practically unchanged even after considerable grinding in contact with water, oxygen, silicates and pebbles. Therefore it would appear unlikely that ferric xanthate could be extracted from pyrite even by extracting the mineral immediately after treatment with the agent. On the other hand, if there is present with the pyrite, at the time the xanthate is made to act on it, a solvent for ferric xanthate in which that substance is protected from decomposition, the prospect for extracting ferric xanthate from xanthate-treated pyrite is much better.

Several types of experiments were conducted. In the first series pyrite was ground, floated by addition in the flotation cell of potassium ethyl xanthate, and the froth caused to fall in a pan full of benzene. The froth was agitated with the benzene, the liquids were separated and the benzene phase was filtered and studied. In the second series pyrite was ground in benzene with potassium n-amyl xanthate and ethyl dixanthogen in the absence of water.(18a) In the third series pyrite was ground with potassium n-amyl xanthate in the presence of water and benzene and in the absence of oxygen. The liquids were separated, and the benzene phase filtered and studied.(18b)

TABLE 13.—*Results of Treating Pyrite in Flotation Cell with Ethyl Xanthate and of Immediately Leaching Mineral for Ferric Xanthate*

Mineral used, grams	500	500	500
Time of grinding, min	50	50	50
Xanthate added (in cell), grams	2.5	2.5	2.5.
Sodium carbonate added (in mill), grams	0.0	1.0	5.0
pH of aqueous solution	7.5	10.0	11.3
Ferric xanthate in benzene solution	none[a]	none[a]	none[a]
Potassium xanthate in tailing solution	none	none	none

[a] Less than 10^{-5} grams, if not absolutely nil.

From the first series no ferric xanthate whatever was extracted (Table 13). This may be explained by assuming decomposition of whatever ferric xanthate is formed in the short time allowed for the reaction (about 2 min.) or by considering that no ferric xanthate did form.

From the mineral in tests of the second series were obtained small amounts of ferric xanthate together with sulfur, and various oils and greasy substances. Failure to obtain ferric xanthate resulted whenever oxygen was present. These results are explainable in part by the extremely low solubility of potassium xanthates in benzene.(8)

TABLE 14.—*Results of Reaction Tests of Pyrite with Potassium n-amyl Xanthate, Ground Together in Presence of Oxygen-free Water and Benzene in Oxygen-free Nitrogen under Slight Pressure*

Mineral Substance Used	Potassium Xanthate Used, Grams	Xanthate Abstracted as Potassium Salt, Grams	Ferric Xanthate Leached from Mineral, by $CuCl_2$ Titration Method,(18 Grams	Iron in Benzene, by Analysis, Grams	Elemental Sulfur in Benzene	pH of Aqueous Solution
Cleaned pyrite	2.0	0.285	0.052			
Cleaned pyrite[a]	2.0	0.640	0.000			8.7
Cleaned pyrite	2.0	0.505	0.024	0.005	0.180	9.7
Uncleaned pyrite	2.0	1.098	0.464	0.063	0.178	9.1
Uncleaned pyrite	0.0			0.000	0.032[b]	5.1
Aplite	0.0			0.000	0.010[b]	7.6
Aplite	2.0	0.260	0.084	0.008		8.6
Cleaned aplite	2.0	0.995	0.432			8.1
Cleaned pyrite	2.0	0.718	0.051	0.018	0.018	8.9

[a] Mill leaked during test.

[b] Possibly leached by benzene from rubber gasket.

From the mineral in tests of the third series ferric xanthate was obtained in small, yet definitely larger amounts than from tests of the second series. Obviously, in spite even of the presence of the benzene as ferric-xanthate solvent and protector, and of the almost complete absence of oxygen, little ferric xanthate is obtained. This may be due to the great facility with which ferric xanthate decomposes. The results obtained are presented in Table 14. In this connection it must be said that ferric xanthate was observed to form in benzene-water-potassium xanthate even if a mineral presumably barren of iron was used (e.g., with aplite). After considerable investigation this was traced to the metallic iron abraded from the grinder during the preliminary comminution of the mineral. Since the amount of ferric xanthate observed to form from aplite is greater than that formed from pyrite, and since the amount formed from acid-cleaned pyrite is vastly less than that from uncleaned pyrite, it is possible, of course, that none of the ferric xanthate observed in connection with pyrite is derived from reaction of that mineral with the alkali xanthate. For the time being extension of the Taggart-Taylor-Knoll theory of a metathetical base-metal xanthate coating on pyrite cannot be considered as proved, even though every opportunity has been given, during experimentation, to such a coating to form, and to not decompose. At any rate, if such a coating does form, it is certain that it has already decomposed by the time the mineral is floating, the decomposition products consisting of ferric hydroxide, sulfur and oils, including the alkyl ester of the alkyl xanthic acid whose alkali salt was used.

SUMMARY

The experimental evidence summarized in this paper as the result of several years work by several investigators is still inconclusive in that it is not possible to generalize as to the mode of action of xanthates on sulfide minerals. It is possible, however, to consider the following as established:

1. The oxidized coatings on galena react metathetically with alkali xanthate in solution in water to produce coatings of lead xanthate.
2. Galena reacts with dixanthogens to yield lead xanthates on the mineral but no ions in solution. This reaction is not of the same type as that covered under the preceding heading.
3. In the reaction between galena and dixanthogen at pH about 7, there is formed a red-brown substance soluble in benzene which breaks down to lead xanthate, sulfur and lead sulfide or to sulfur and lead sulfide.
4. Lead xanthate formed as a coating on galena decomposes on prolonged grinding to yield sulfur and an oil or mixture of oils of which the exact composition has yet to be determined.
5. The oxidized coatings on chalcocite react metathetically with alkali xanthate in solution in water to produce coatings of cuprous xanthate.
6. Chalcocite reacts with dixanthogen to yield cuprous xanthate.

7. Cuprous xanthate at the surface of chalcocite, and because of the action of hydroxyl ion and/or of oxygen in water decomposes in steps yielding a number of definite intermediate products, which may be regarded as consisting of Cu_2S or CuS coordinated with cuprous xanthate, and/or with cuprous thiocarbonate. These intermediate products contain more and more Cu_2S or CuS as the decomposition proceeds.

8. Neither sulfur nor oil has been identified from leach products of treated chalcocite.

9. Covellite reacts with dixanthogens to yield cuprous xanthate and sulfur; with alkali xanthates likewise to yield cuprous xanthate and sulfur, after passing through an intermediate stage in which dixanthogen is produced.

10. Unless extreme care is exercised to extract ferric xanthate immediately upon formation, so as to prevent its decomposition, that compound is not observed in the reaction of pyrite with xanthates. Instead, oils and sulfur are recovered. What ferric xanthate can be extracted from pyrite may not originate from the pyrite.

11. It has yet to be established what the reaction is that occurs between pyrite and dixanthogen.

From this summary and from other evidence, it is clear now that the early ideas of adsorbed reversible coatings on sulfide surfaces must be given up. Instead, it is necessary to view the sulfide not only as capable of reacting through its oxidized coatings with the xanthate ion in solution but also directly with minute droplets or micelles of dixanthogen. Since, furthermore, xanthate ion oxidizes readily to dixanthogen, in the presence of oxygen xanthates may first go to dixanthogen, and the latter may react with the mineral; or the combined reaction of xanthate, the sulfide mineral and oxygen may take place at the surface of the mineral. In most if not in all cases the coating formed must be very thin; perhaps, indeed, it is a monomolecular coating. This formation is not a reversible phenomenon, but rather a step in a general evolution of the surface of the mineral, which under practical conditions ever fails to reach equilibrium. Either through the action of oxygen or through that of hydroxyl ion, or through an internal molecular rearrangement, there is brought about a gradual change in the chemical entity of the coating, which eventually becomes a sulfide (galena, chalcocite or covellite) or a hydroxide (pyrite) of the metal in the mineral together with sulfur and oils.

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The Case for the Chemical Theory of Flotation

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In a previous paper[1] in which one of the authors collaborated, it was postulated that *All dissolved reagents which, in flotation pulps, either by action on the to-be-floated or on the not-to-be-floated particles affect their floatability, function by reason of chemical reactions of well recognized types between the reagent and the particle affected.* This generalization, apparently, like many pioneer structures, had for certain observers a flimsy appearance, and these, judging its stability rather by the number than by the character of its foundation posts, have attempted variously to overthrow it, either by the frontal attack of general denial, or by the flanking movement involved in assuming that those cases in which the facts were unknown or unproved were, therefore, discordant. But as time has passed and further evidence on the question has accumulated, it has become apparent that the results of verifiable experiments add stone by stone to the substructure of the hypothesis, and that the supertructure has lost nothing in weathering. The present paper constitutes an examination of the opposing positions in the light of the more recent evidence developed.

It will clarify the discussion if, at this point, we state more fully the implications of the disputed hypothesis. In the first place, it is explicitly limited to reagents that are dissolved in the water of the flotation pulp. Next, it refers only to such of these dissolved reagents as act on the solid particles in the pulp to affect their floatability. This includes the collectors, activators, and certain depressors and gangue-control reagents, but not the pure frothers, and not, necessarily, conditioners whose function seems to be merely that of pH control, although in certain cases it has been demonstrated that such control has the effect of regulating the primary reaction of the collector, depressor, or the like. Finally, there is no limitation as to the nature of the chemical reaction postulated.

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[1] Taggart, Taylor and Knoll: Chemical Reactions in Flotation. *Trans.* A.I.M.E (1930) **87**, 217.

Metathesis is the type most commonly met with and most thoroughly investigated, but oxidation and reduction reactions have been established both in conditioning and collection, and addition reactions are to be suspected in the case of certain of the nitrogenous collectors.

Chemical reaction between certain inorganic flotation agents in solution and the surfaces of certain of the pulp minerals was postulated as early as 1904.[2] Christensen,[3] in 1918, discovered that only those oils were useful in floating oxide minerals that contained an ingredient (a fatty or a resin acid) which reacted with the mineral to be oil-coated. As early as 1925 it was generally accepted that conditioning of sphalerite with copper sulfate involved a reaction producing a surface coating of copper sulfide on the sphalerite, and probably a majority of investigators since that time have believed—although with very little supporting evidence adduced until recently[4]—that activation and depression functioned through metathesis between reagent and affected mineral. But during this same time the action of the soluble collectors was attributed to adsorption, and this stand persists to the present day.[5]

The attitude of the more rational exponents of the adsorptionist school may be gathered from the following citations: Wark and Cox[6] say:

> . . . We find that there is a strong connection between adsorption of xanthates and the solubility of the heavy-metal xanthates, but we are not yet able to decide whether this amounts to an identity. Oil flotation definitely does not follow a similar course. Many cases have been cited which are apparent exceptions to the chemical theory of adsorption, and until these have been investigated, no further progress will be possible.

Somewhat later, Cox and Wark[7] summarize their stand as follows:

> . . . . No factor is more important in flotation by soluble collectors than adsorption, and this is particularly true of adsorption at the solid surface. . . . Under certain conditions unimolecular films are adsorbed by the minerals which then display a definite air avidity.
>
> . . . . A valid explanation for the adsorption of xanthate by minerals would mark an important step forward in the theory of adsorption.
>
> The balance of the evidence appears to be against the chemical theory and for the present it is suggested that in its place there should be substituted the generalization "The ability of a mineral to adsorb a soluble chemical flotation collector containing S is closely related to the solubility of the salt formed by the collector and the metal of the mineral."

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[2] De Bavay: U. S. Patent 864597.

[3] Christensen: U. S. Patent 1467354.

[4] Wark and Cox: See pages 245, 267, this volume.

[5] Cox and Wark: *Jnl. Phys. Chem.* (1933) **37**, 797.
Gaudin: Flotation. New York, 1932. McGraw-Hill Book Co.
Ostwald: *Kolloid Ztsch.* (1932) **58**, 179; **60**, 324.
Holman: Inst. Min. and Met. *Bull.* 314 (1930).

[6] Wark and Cox: Page 228, this volume.

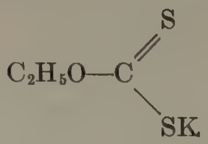
[7] Cox and Wark: Reference of footnote 5.

Gaudin[8] says:

> Collecting agents (excluding so-called "collecting oils") may be classed in two varieties: those that form definite compounds by metathesis with the surface of the minerals, and those that do not seem to do so. The mode of operation of the latter type of collecting agents is not entirely clear yet it is established that removal of the reagent from solution takes place; it is not known whether the reagent simply adheres to the surface of the minerals or is changed at the surface to a different chemical form.

As evidence against metathesis Gaudin cites the case of xanthate and pyrite, arguing that, although pyrite abstracts xanthate ion from solution, ferrous xanthate is relatively soluble; ferric xanthate does not form with ferric ion; siderite is not floated by xanthates, even in high concentrations; neither ferrous oxides, hydroxides or carbonates abstract xanthate ion from solution; and badly oxidized pyrite is difficult to float with xanthate unless first sulfidized. He further is in doubt as to the action of xanthates with chalcocite and activated sphalerite.

Ostwald[9] sets up a peculiarly fantastic development of the adsorption theory for soluble collectors in which potassium ethyl xanthate, for example, conceived of as a sort of a three-legged molecule, first adsorbs at the surfaces of the bubbles and the sulfide particles in the pulp, and then, when a bubble and a mineral particle meet, the particle penetrates the bubble wall, drying immediately on that part extending into the gas cavity of the bubble, and the three-legged molecules simultaneously jump into position along the line of three-phase (air-solution-solid) contact, with one leg extending into the gas, one into the liquid and one against the solid phase. Thus oriented these molecules are supposed to exert a restraining influence on the movements of the phases, resulting in what Ostwald calls *adlinear* flotation. In potassium ethyl xanthate, the structural formula for which is



Ostwald conceives the ethoxy group as air-avid, the double-bond sulfur as metal-avid, and the SK group as water-avid.

This theory seems to have been proposed to account for a belief, founded on a calculation by Gaudin,[10] that the amount of soluble collector used in modern practice is insufficient to form a film on all of the sulfide floated. Ostwald calculates that this same amount, stretched out as a line, would serve. No experimental basis is offered for the theory, and the only inferential basis seems to be the calculation referred to.

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[8] Gaudin: Reference of footnote 5.

[9] Reference of footnote 5.

[10] Gaudin: Univ. of Utah *Tech. Pub.* 1 (1928) 18.

The theory overlooks the fact that the alkaline xanthates are not surface active,[11] which would seem to bar adsorption at the air-water interface; that they ionize substantially completely in solutions of the strength used in flotation; that the ion of the alkali metal does not go with the mineral, but remains behind in the solution, thus proving that the molecule does not act as a unit; and that when xanthate ion is abstracted from solutions by sulfides, the extent of abstraction—all other conditions being equal—is proportional to the sulfide surface exposed to the solution.

This Ostwald theory is so fantastic that mention would have been completely omitted here except for the wide publicity that it has been accorded. If the discussion anticipates by a few pages the development of the experimental facts that it asserts, it is because of a desire to wind up the matter as quickly as possible.

Ravitz and Porter[12] attack the chemical theory from a different angle. They describe an investigation of the flotation behavior of "oxygen-free galena." They devised extremely ingenious apparatus in the attempt to insure an oxygen-free flotation atmosphere, and a much less convincing method for producing a "pure unoxidized galena" surface.[13] They

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[11] DeWitt and Roper: *Jnl.* Amer. Chem. Soc. (1932) **54**, 444.
DeWitt and Makens: *Jnl.* Amer. Chem. Soc. (1932) **54**, 455.

[12] Ravitz and Porter: A.I.M.E. *Tech. Pub.* 513 (1934).

[13] The authors state that after soaking the galena for their "oxygen-free" tests in saturated ammonium acetate solution for at least a week, in order to dissolve lead sulfate from the particle surfaces, they then attempted to wash out the resulting ammonium sulfate-lead acetate solution by displacement washings with oxygen-free ammonium acetate followed by oxygen-free water. But at the end of their washing period they still, according to their own statement, obtained a test for acetate by the cupric chloride-sodium chloride method. Assuming, as experience would indicate that the last acetate ion would come from the liquid nearest to the solid surface, since this is the most difficult to displace by washing, then, since this is also the region of highest concentration of lead, we may assume further that lead acetate is the carrier of this last acetate ion. We must, therefore, conclude that there was still lead acetate in the immediate vicinity of the galena-particle surfaces when the washing ended and the "oxygen-free" experiments started. Taylor and Knoll (p. 382, this volume) have shown that the solubility of lead sulfate at galena-particle surfaces in water is considerably less than that of bulk lead sulfate in water; i.e., that an equilibrium is established between undissolved lead sulfate at a galena surface and water, at a concentration in the water well below the solubility product of lead sulfate. We may expect, therefore, that in the Ravitz and Porter experiments there was still some lead acetate—which is to say, soluble lead—at the surface of the galena particles when these experimenters made their flotation tests with xanthate, and that the xanthate reacted with this soluble lead surface to form lead xanthate in the usual fashion.

A further consideration, admittedly speculative, should none the less be mentioned here, as it has a bearing on all experiments in which it is attempted to clean and purify ground galena by washing with ammonium acetate. Aged ground galena such as Ravitz and Porter used contains a relatively large amount of lead carbonate. Treatment with ammonium acetate brings together the three ions; viz., lead, carbonate found that when they used what they conceived to be pure unoxidized galena in their oxygen-free apparatus, they were able to effect flotation with no added reagent. They concluded that their experiments supported the old idea of inherent floatability of certain minerals, reasserted by Sulman[14] in 1930, and in highly tentative fashion they suggested that the function of xanthate is

> . . . in part, that of a cleansing agent; i.e., to remove oxidation products from the surface of the mineral, leaving the nonpolar sulfide free for the displacement of water by gas.

Another group of investigators, among whom may be numbered Takakuwa[15] and Ralston,[16] follow Sulman[17] in ascribing flotation to differential flocculation of the particles to be floated, without examining into the mechanism that controls flocculation. Since, however, this puts the cart before the horse, in that mineral flocculation is an effect of the preceding treatment and a step consequent thereon on the way to flotation, and since the chemical theory deals with the mechanism of the antecedent step, the beliefs of this school need be regarded neither as *pro* nor *con* in the argument on the validity of the theory.

Argument for the Theory

Since the bulk of the opposition to the chemical theory is represented in the adherents to the adsorption hypothesis, we can best clear the decks for action on the argument if we examine into just what is denoted and connoted by the word adsorption. The word is a stand-by in the literature of colloidal chemistry, used to clothe a large variety of unrelated and generally misunderstood phenomena with an air of knowledge and understanding. Correctly defined and used the word has its place in scientific literature. It then denotes a state of difference of concentration of a solute at the boundaries of a solution from the concentration of the same solute in the bulk of the solution; an excess or deficit as the case may be. It denotes a concentration difference and nothing more. It

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and acetate, which are intentionally brought together in the Dutch process for making white lead (basic lead carbonate). It is not straining speculation, therefore, to assume that this highly insoluble lead compound was formed at the galena surfaces in the Ravitz and Porter acetate washing and was not removed to any appreciable extent by the subsequent water washing. When white lead is shaken in xanthate solution it becomes distinctly yellowish by reason of precipitated lead xanthate. Hence any of this compound, like the residual acetate, would enter into the chemical reaction whereby the galena-particle surfaces become xanthate coated.

[14] Sulman: Inst. Min. and Met. *Bull.* 311 (1930).

[15] Takakuwa: *Proc.* World Eng. Congress, Tokyo, 1929; Min. and Met., pt. 3, 395.

[16] Ralston: *Trans.* A.I.M.E. (1930) **87,** 247.

[17] Sulman: *Trans.* Inst. Min. and Met. (1919) **29,** 44; also reference of footnote 14.

does not tell anything as to the state of the concentrated solute, or of the mechanism of concentration, or of the condition of solute (or solvent) in the boundary layer, or of the state of boundary surface of the adjacent phase. It has somewhat the factual definiteness in the literature of chemical and physical science that the conception of heaven has in religious thought. Yet—for this very reason, perhaps—it is commonly used as a haven of refuge by writers lost in a morass of shaky experiment and muddy thinking.

The fundamental conception of adsorption is set forth in the famous Gibbs[18] equation:

$$U = -\frac{c}{RT} \cdot \frac{d\sigma}{dc}.$$

in which U is the excess of concentration in the boundary layer over that in the bulk solution; R is the gas constant; T, the absolute temperature; c, the concentration of solute in the bulk of the solution; and σ, the surface tension. This equation states that when the change in surface tension with change in concentration of the solution is negative (i.e., when surface tension decreases as concentration of solute increases), the excess of solute in the surface layer is positive; in other words, that the concentration of solute in the surface layer exceeds that in the bulk, and vice versa. This formula has a sound thermodynamic basis and is generally accepted. Its prediction has been proved qualitatively in a large number of cases of gas-liquid and liquid-liquid interfaces. On the other hand, quantitative confirmation, even for gas-liquid interfaces, is lacking, and such quantitative work as has been done points rather to a boundary zone of intermediately variable concentration than to a boundary surface sharply differentiated in concentration from the bulk solution.

In flotation, frothing distinctly involves simple molecular adsorption. That is to say, the liquid film of each bubble has, at its contact with the contained gas, a higher concentration of the molecules of the frothing agent than is present in the bulk of the solution—or even in the interior of the film. Miss Benson's[19] famous experiment with amyl alcohol, which is the classical historical qualitative demonstration of Gibbs' theorem, was performed by frothing an amyl alcohol solution, separating the froth, and, by analysis, showing a higher concentration of amyl alcohol therein than in the residual solution. Many similar experiments on other surface-active materials, including flotation frothers,[20] have been reported.

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[18] Gibbs: Collected Works, **1**, 233, New York, 1928. Longmans, Green and Co.

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[20] Fahrenwald: *Min. & Sci. Pr.* (1921) **123**, 227.
Fahrenwald: *Trans.* A.I.M.E. (1924) **70**, 647.
Taggart and Gaudin: *Trans.* A.I.M.E. (1923) **68**, 479.
Taggart, Taylor and Ince: *Trans.* A.I.M.E. (1930) **87**, 285.

Excellent qualitative evidence of the extent to which concentration goes in a bubble film may be obtained by introducing some powdered Sudan III dye, which is fat-soluble but water-insoluble, into a dilute solution of amyl acetate in a square glass jar and then stirring, first gently and thereafter violently. Before and during the gentle stirring no color is imparted by the dye to the water, and individual particles examined under the microscope are blood red and show sharp edges and corners characteristic of nonsolution. After violent stirring, however, on stopping the agitator, a pink froth collects, the pink color being characteristic of a solution of Sudan III in amyl acetate. The indication, of course, is that adsorption of amyl acetate in the bubble films has gone forward to such an extent that, in this respect at least, the concentrated liquid acts as relatively pure amyl acetate.

When, however, it is attempted to apply the adsorption hypothesis to the phenomena of conditioning and collection, the investigator is faced by the limitation that the interfaces involved are those between a solid and a solution, and difficulty is at once encountered in the fact that no experimental means is known to measure the tension at solid boundaries. Hence neither prediction from nor agreement with the Gibbs equation can be properly asserted, as neither σ nor $d\sigma$ is determinate. Experimental data are recorded in plenty in the literature of colloidal chemistry indicating concentration of solutes at solution-solid interfaces, from which it is inferred—on the assumption that the Gibbs formula applies—that the solute lowers the solution-solid interfacial tension. Thereafter—such is the viciousness of circular reasoning—the inference is accepted as fact and it is further concluded that adsorption therefore occurs because the solute lowers the solution-solid interfacial tension. But, until some independent proof of the assumption itself is brought forward—and none has appeared to date—the inference, and the applicability of the Gibbs equation must both be recognized to lack experimental verification completely. Adsorption at solid surfaces—properly defined and clearly understood to denote nothing more than concentration of solute (*or of a reaction product of the solute*)—may be accepted as well established fact, but to attribute to the term adsorption thus applied any element of explanatory character is to adopt the mental attitude of the ostrich seeking concealment.

The chemical hypothesis of conditioning and collection, on the other hand, takes the clear-cut position that the abstraction of solute from flotation-pulp solutions by mineral particles is the result of chemical reactions between the solute and the surface material of the solid particles. It does not deny the possibility that molecular adsorption of certain solutes at certain solid surfaces may be an experimental fact in colloidal chemistry. It takes no position on this score at all. But it does assert that, in so far as verifiable experimentation goes, the evidence all points to the conclusion that in collecting and conditioning in flotation, alteration of the mineral-particle surfaces is not of this character.

In an extremely careful and complete piece of research work, A. F. Knoll[21] followed through quantitatively the reactions between pulverized galena and potassium ethyl xanthate solutions. He found complete stoichiometric balance between xanthate ion abstracted from solution and sulfate plus reduced divalent sulfur-oxygen ions thrown into solution, when the concentration of the original potassium ethyl xanthate solution was 16.3 mg. per liter and less, which is of the order of concentration of the collector solutions usually employed in flotation. With higher concentrations of xanthate than those usually employed in the concentration of sulfide ores he found that more xanthate ion was removed and that a corresponding amount of carbonate ion went into solution, still maintaining the stoichiometric balance. The extent of this xanthate-carbonate interchange increased with increasing xanthate concentration, with given batches of galena, until a constant point, representing the total carbonate initially present in the galena, was reached. The amount of sulfate and reduced sulfur-oxygen ions displaced increased with the age of the galena after grinding, as did also the ratio of sulfate to the reduced sulfur-oxygen ions, thus proving that oxidation toward sulfate was progressive in the laboratory atmosphere, but that oxidation to carbonate did not occur under the same conditions He further showed that sulfide ion did not enter into the xanthate metathesis, and he confirmed the observation that the metathetic reaction left the alkali ion of the xanthate used unchanged in concentration in the solution.

Some later work by Gaudin and coworkers[22] indicates that when xanthate solutions are contacted with galena for excessive times, and when the concentrations of xanthate are far in excess of those used to float galena, dixanthogen is formed, and they postulate that this oxidation product of xanthate may be the effective agent in the flotation of the galena. But the work of Wark and Cox,[23] amply verified in the laboratory at Columbia, shows sharp differentiation between the xanthate and corresponding dixanthogen contact angles, and further that galena in contact with a given xanthate solution, under conditions of concentration, temperature, and solution atmosphere reasonably paralleling those in the mill, gives a contact angle characteristic of the particular xanthate ion.

It would seem, therefore, that the chemical hypothesis is reasonably completely substantiated for galena and ethyl xanthate. Yet the adsorptionists would have us believe that either the potassium ethyl xanthate molecule, as a whole, is held oriented at the galena surface by some unexplained mechanism, or that the ethyl xanthate is held through "unsatisfied and stray valences" and the like. But their first hypothesis is upset completely by the fact that when pulp solution is filtered away from galena, the potassium ion remains in solution and goes with the filtrate; while the second hypothesis fails to meet the fact that crystalline lead xanthate can be recovered from the galena surface by a nonaqueous solvent; e.g., ethyl alcohol, as well as that each ion of xanthate taken up and held by the galena displaces its stoichiometric equivalent of other ions from the galena surface. If these facts be evidence *pro* physical adsorption of the type of Gibbs and *contra* metathesis, the common foundations of analytical chemistry would seem to be footed in quicksand.

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[21] Knoll: Dissertation for Doctorate, Columbia University (1932); also Taylor and Knoll: page 382, this volume.

[22] Gaudin and Wilkinson: *Jnl. Phys. Chem.* (1933) **37,** 833.
Gaudin and Malozemoff: page 303, this volume.

[23] Wark and Cox: page 189, this volume.

The action of other xanthates than ethyl with galena has not been followed through completely. It has been shown semiquantitatively at Columbia that both amyl and butyl xanthate ions are abstracted from solution by galena without corresponding abstraction of potassium ion, and that oxygen-sulfur ions are displaced from the galena surface. These experimental facts confirm the reasoning from analogy, based on the usual similar behaviors of homologs in organic chemistry, that metathesis likewise underlies the action of other xanthates.

The authors have verified the work of Luyken and Bierbrauer,[24] using, however, sodium oleate instead of the stearate. The procedure involved shaking deslimed galena intermittently for one hour in a 1:3 pulp containing the oleate, settling, decanting, elutriating the solid to free it of slime and free precipitated soap, and then analyzing the products as follows:

A 100-c.c. portion of the decanted pulp liquor was acidified with 5 c.c. of conc. HCl, extracted three times with alcohol-free ether (30, 10 and 10-c.c. portions respectively), the combined ether extracts were evaporated to dryness in a tared beaker and the residue weighed as oleic acid.

Another aliquot of the pulp liquor was analyzed for sodium by a modification of the sodium zinc uranyl acetate method.[25]

The washed galena was placed in a dilute solution consisting of about 300 c.c. water and 4 c.c. conc. HCl, and the mixture was then extracted with ether as above The oleic acid content of the ether was determined, as was also the sodium content of the aqueous extract.

A blank test for sodium was run on a sample of the original galena and tests for sodium and oleate ions were made on the original soap solution.

The results of two test series, one with sodium carbonate used as a conditioning agent and one without, are given in Table 1. It is apparent that sensible and unmistakable quantities of oleate ion were taken out by the galena, while within the limits of experimental error no sodium ion was so removed. Calcite and apatite similarly abstract oleate ion from soap solutions, but quartz does not.

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[24] Luyken and Bierbrauer: *Mitt.* K. W. Inst. f. Eisenforschung (1929) **11,** 37.

[25] See Dobbins and Byrd: *Jnl.* Amer. Chem. Soc. (1930) **53,** 3288.
Barber and Kolthoff: *Jnl.* Amer. Chem. Soc. (1928) **50,** 1625.
Pulman: *Amer. Jnl. Sci.* [4], **16,** 229, and bibliographies listed therein.

TABLE 1.—*Results of Two Test Series*

Substance	Introduced, Mg.				Recovered, Mg.		
	Original Soap Solution	Original Na_2CO_3 Solution	Galena Blank	Total	Pulp Liquor	Galena	Tota
Sodium	2.94		0.22	3.16	3.08	0.25	3.33
Oleic acid	31.8			31.8	26.3	4.8	31.1
Sodium	2.86	32.80	0.22	35.88	35.75	0.25	36.00
Oleic acid	32.3			32.3	24.9	6.1	31.0

In an attempt to answer the points raised by Gaudin[26] regarding the behavior of pyrite and other iron compounds, the authors have investigated the action of pyrite with ethyl xanthate, with the following results.

1. We confirm the ethyl-xanthate contact angle reported by Wark and Cox.[27] This indicates, since each xanthate (and each other organic radical tested by them) has a definite and characteristic contact angle, that the xanthate, and not a decomposition product of the xanthate ion, is the effective collector.

2. Pyrite abstracts the ethyl xanthate ion from aqueous solution, but not sodium ion. This parallels the action of ethyl xanthate with galena, and all of the reasoning founded on these facts and argued with respect to galena is applicable in the case of pyrite.

3. We confirm Gaudin's observation that ferrous xanthate is relatively soluble in water. If a dilute solution of a ferrous salt is added to a xanthate solution of such strength that the resultant xanthate concentration is 25 mg. per liter, no precipitation occurs at first. If oxidation is permitted, however, a brownish white cloudiness appears. This is probably due to a mixture of ethyl dixanthogen and ferric xanthate.

4. We do not confirm Gaudin's statement that ferric salts with ethyl xanthate do not yield ferric ethyl xanthate. On the contrary, we find that when an aqueous solution of a ferric salt is added to an aqueous ethyl xanthate solution, a heavy chocolate-brown flocculent precipitate is formed. This is soluble in ether and the ether extract yields on analysis iron and ethyl xanthate ions in the ratio of 1:3 (experimentally, 1:3.06 and 1:2.89 on two trials), which is to say, ferric xanthate. Careful

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[26] Gaudin: page 341, this volume.

[27] Wark and Cox: page 200 this volume.

evaporation of the ether solution yields a black crystalline mass which analyzes for iron and xanthate ions in the ratio 1:2.71. Under the microscope the mass shows rust-brown impurities, which would account for the excess of iron and which probably result from decomposition of ferric xanthate during evaporation of the ethereal solution. A batch of crystals, after standing one week in a desiccator, had a strong odor of dixanthogen. A very dilute acetone solution of the crystalline material yielded blood-red hexagonal plates on slow evaporation. Similar crystals are obtained from acetone extracts of xanthate-treated pyrite.

5. We have found that aqueous extracts of air-ground pyrite contain sulfate, ferrous and ferric ions.

6. If a pyrite particle with freshly polished surface is conditioned for an hour in a solution of 2 grams per liter of sodium sulfite and the solution is then made up to a concentration of 25 mg. per liter of potassium ethyl xanthate and a contact-angle test is made, the reading is zero—no contact. Sodium sulfite has reduced ferric to ferrous ion. It has no effect in a similar test with galena.

These tests would seem to prove that the action of ethyl xanthate with pyrite involves: (1) oxidation of the pyrite surface to ferric sulfate by oxygen from the pulp or by some oxidizing salt in solution; (2) metathesis of this ferric salt at the particle surface with the ethyl xanthate salt to form a precipitate of ferric xanthate. They would also indicate that pyrite flotation with xanthates will be aided by a conditioning treatment that accelerates oxidation of the pyrite surface, and, conversely, that a strongly reducing solution will depress pyrite when a xanthate collector is used.

We have confirmed Gaudin's statement[28] that xanthate ion is abstracted from solution by sphalerite preconditioned in copper sulfate solution; likewise by chalcocite. We find further that with both of these minerals, when sodium ethyl xanthate is used sodium ion is not abstracted by the mineral.[29] Both minerals were found by Wark and Cox to give characteristic xanthate contact angles and we have verified this.

We fail to be impressed by the argument[30] that the high solubility of copper sulfate in water and the low solubility of cupric sulfide make it impossible to conceive that copper xanthate can be the collector film on copper-conditioned sphalerite. It is the common experience of analytical chemists that freshly precipitated copper sulfide oxidizes very readily; so easily and quickly, in fact, that appreciable amounts of soluble copper are carried through paper on which such a precipitate is being filtered and washed. Hence, as to this objection, the surface of copper-conditioned sphalerite, highly insoluble cupric sulfide though it may be, is readily attacked and rendered soluble in an oxygenated pulp.

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[28] Gaudin: Reference of footnote 5, 66.

[29] The experimental figures are: Starting with a sodium ethyl xanthate solution containing 4.3 mg. sodium per 100 c.c., the filtrate after contact with chalcocite contained 4.2 mg. sodium per 100 c.c.; with sphalerite conditioned for 15 min. in a solution containing 100 mg. per liter of $CuSO_4.5H_2O$, and then filtered off and treated with the xanthate solution the sodium residue in the filtrate was 4.3 mg. per 100. cc.

[30] Gaudin: Reference of footnote 5, 66.

The mechanism of anchoring the copper ion from a molecule of highly soluble copper sulfate at the surface of the particle on which it forms by oxidation is somewhat more difficult to picture. But the evidence that surface copper sulfate, soluble though it may be, is not immediately dislodged by water, is the same as with lead sulfate on galena, but more striking; a water extract of an oxidized copper sulfide surface (air-ground chalcocite) carries a rather small amount of sulfate ion; the same material, leached by xanthate solution for the same time, throws off a tremendously increased quantity of sulfate ion, which must come, clearly, from anchored copper sulfate.

Before leaving the subject of the xanthate collectors, we wish to discuss briefly the significance of the adsorptionist viewpoint embodied in the following quotation from Gaudin:[31]

> The mechanism by which xanthates float other sulfides than galena may involve . . . an adsorption of xanthate ions without further reaction.

It is inherent in the whole conception of ions that they are completely soluble in and miscible with the medium in which ionization occurs. In fact, our best mental picture of an ionizing liquid is of a substance that tears a molecule apart by reason of affinities for its ion constituents. Hydration of ions in aqueous solutions would seem to indicate greater affinity between water and the ion than between the ion and its oppositely charged companion. It follows, then, unless the ion is completely changed by its postulated adsorption, that it must retain its water-avid character after adsorption. Under such circumstances it is difficult to see how it can impart the character of water repellency to the sulfide surface on which it is supposed to adsorb "without further reaction."

Almost the last stronghold of the adsorptionists falls, if Christensen's hypothesis with respect to the action of collecting oils[32] can be verified. Thus Wark and Cox in the citation at page 349 say, in effect: Even though there may be some doubt as to whether xanthates act by metathesis, oils definitely do not. Gaudin (see p. 350) explicitly excludes oils from his classification of chemical collectors. But Nujol, which is a highly purified mixture of saturated paraffin (and possibly naphthenic) hydrocarbons, definitely freed of organic compounds of acidic character, does not make contact in the bubble machine with grease-free polished surfaces of galena, sphalerite, pyrite, apatite or calcite. When contaminated with a minute amount (0.05 per cent) of oleic acid, however, it does make contact. If the minerals are preconditioned with sodium oleate, pure Nujol makes contact. If they are preconditioned with potassium ethyl xanthate, the pure Nujol makes contact with galena and with pyrite but not with sphalerite and the nonmetallics. This means clearly that it is first essential for oil wetting, just as for air wetting, in the presence of water, that the mineral particle to be wetted be first coated with an organic hydrocarbon-type surface. Whether the preferential wetting of such a surface by oil is a Gibbs adsorption phenomenon, i.e., occurring because a reduction of the total surface energy of the system is thereby effected, or whether the mutual miscibility of the hydrocarbon-like surface of the conditioned mineral and the hydrocarbon oil is the seat of the driving force, is neither proved nor apparent. But for the purposes of the present argument the answer to this question is immaterial. The significant fact is that even oiling by collecting oils must be preceded by the formation on the mineral particles of an organic surface film which, in the two cases tested, at least, is formed by metathesis.

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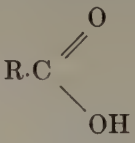
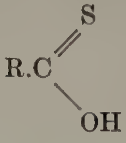
[31] Gaudin: Reference of footnote 5, 67.

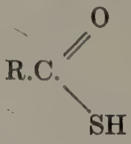
[32] Christensen: Reference of footnote 3.

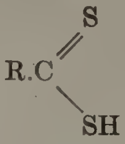
As a practical guide in experimentation with flotation collectors, it may be pointed out that the great majority of effective chemical collectors are organic acids, or their salts (usually those of the alkali metals) or compounds of distinctly acidic character. Thus oleic acid, $C_{17}H_{33}.COOH$ is typical of the entire fatty acid group with the general structural formula



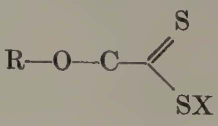
in which R is any hydrocarbon radical, saturated or unsaturated, and COOH is the characteristic carboxylic acid radical. Starting with such an acid, it is possible to substitute sulfur for either or both of the oxygens of the carboxyl, thus obtaining compounds of the types



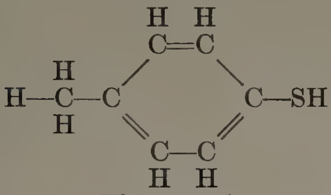


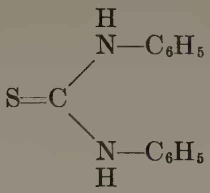


the mono- and di-thio acids respectively. The xanthates are salts of a dithio acid, in which, however, oxygen is the link between the hydrocarbon radical and the carbon of the acid, thus

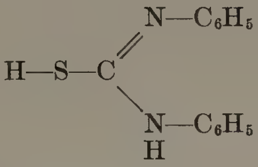


X being H in xanthic acid or an alkali metal in the usual xanthate collectors. In thio-cresol and thio-carbanilid the characteristic acid grouping, —CXYH, is apparently missing, thus:


Thio-cresol


Thio-carbanilid

But if the formula for thio-carbanilid, for example, is written in the tautomeric form,



which experiment shows is ordinarily present in equilibrium quantities with the first form, it is apparent that in both thio-cresol and thio-carbanilid we have the SH group attached to a carbon atom, which, in turn, links it to the rest of the molecule, as is the case in the acids. This does not, in itself, establish acidic character, but further examination of the structures of these two molecules shows that, if we begin writing the formulas, starting with the H attached to the S, and continue we have $\underset{1}{H}—\underset{2}{S}—\underset{3}{C}=\underset{4}{C}$ for the thio-cresol and $\underset{1}{H}—\underset{2}{S}—\underset{3}{C}=\underset{4}{N}$ for the tautomeric form of thio-carbanilid. According to Vorländer,[33] a hydrogen atom in a position in the molecule such as is illustrated, that is, such that the linking between the third and fourth atoms removed is a double bond, ordinarily displays acidic character; i.e., is more or less readily replaced by metals. Inspection of the typical carboxylic and thio-acid formulas given shows the acidic hydrogen to be of this so-called Vorländer type. Ordinarily, when O or S is the atom on the 4-side of the double bond, and not bonded on the other side, the acidic character of the Vorländer hydrogen is greater than when some other element occupies the 4-position.

Direct evidence of the strongly acidic character of thio-cresol is found in the fact that it readily forms precipitates with salts of base metals in aqueous solution; e.g., yellow lead thiocresylate. Others of the mercaptan (R.SH) class act similarly.

In this connection, and also in line with a suggestion made in an earlier paper[34] that the utility of a given organic compound of the correct structural formula as a collector for a given mineral might be determined by observing whether a precipitate formed on mixing aqueous solutions of the compound and a soluble salt of the metal of the mineral, an extensive series of tests of this character was run. The early results were disappointing in that precipitates failed to form in many instances in the case of substances known to be collectors. This failure was found, however, in most cases, to be due to determinate factors; e.g., wrong pH of the solution, low concentration of the organic compound due to low solubility, decomposition of the organic compound through oxidation, etc. A summary of the more significant results follows:

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[33] Vorländer: *Ber.* (1901) **34,** 1633.

[34] Taggart: *Jnl. Phys. Chem.* (1932) **36,** 130–53.

Potassium Ethyl Xanthate.—Using solutions of unknown but much higher concentrations both of xanthate and of metal ions than are met with in a flotation operation, Ni^{++} gave a yellowish brown precipitate that changed to reddish brown on standing; Co^{++}, greenish black precipitate; Cr^{+++}, no precipitate at first, a distinct whitish cloudiness (probably ethyl dixanthogen) on standing; Sn^{++}, pale yellow precipitate; Cd^{++}, heavy flocculent clear white precipitate; Al^{+++}, no change at first but a whitish haziness after standing for some time; U^{++++++}, solution changes from yellowish to yellow-brown but remains clear; V^{+++++}, no change; Au^{+++}, pale yellow precipitate; Pt^{++++}, orange precipitate; Bi^{+++}, canary yellow precipitate; Mn^{++}, no change at first, on acidification a white precipitate; Fe^{++}, a faint brownish white cloud at first, becoming more pronounced on standing; Fe^{+++}, a heavy chocolate-brown precipitate; Cu^{++}, a heavy deep yellow precipitate; Pb^{++}, a heavy light yellow precipitate; Zn^{++}, a heavy flocculent white precipitate. With solutions of known and controlled strength, potassium ethyl xanthate in excess of stoichiometric proportions precipitates Co, Ni, $Fe^{(+++)}$ and Ag at 40 mg. per liter concentrations from solutions of their salts with strong-acid ions, but not at concentrations of 1 mg. per liter; Pb is precipitated at 1 mg. per liter but not at 0.5 mg. per liter; Cu at 0.5 mg. per liter. In alkaline-buffered acetate solution lead was precipitated at a concentration of 0.06 mg. per liter; results were doubtful with copper at concentrations below 0.5 mg. per liter, since the turbidity that appeared could not be differentiated from possible dixanthogen. Zn failed to precipitate in stoichiometric mixtures containing 250 mg. of zinc per liter but did with 500 mg. per liter.

Thio-cresol.—A saturated aqueous solution of thio-cresol failed to produce precipitates with the following metals in solutions of their salts with strong-acid ions of metal concentrations of 100 mg. per liter: Co, Ni, Fe, $Fe^{(+++)}$, Pb, $Cu^{(++)}$ and Ag. A yellow colloidal precipitate formed slowly with a lead acetate solution containing 40 mg. per liter of lead, buffered with sodium acetate. Alcoholic solutions of thio-cresol or the solid crystals added to either buffered or unbuffered lead acetate solutions forms heavy precipitates. Precipitation from alkali-buffered solutions with alcoholic thio-cresol (probably in excess of stoichiometric quantities) was effected with the following metals at the concentrations set down: Pb, 1 mg. per liter; Cu, 1 mg. per liter; Co, 5 mg. per liter; Ni, 2 mg. per liter. The alcoholic thio-cresol, in stoichiometric amounts, precipitates Cu, Co, Ni, Pb and Zn from alkali-buffered solutions containing 20 mg. of metal per liter. Cu and Pb are most readily precipitated and Zn least.

Mercaptans.—The acetates of cobalt and copper, in concentrations of 40 mg. of metal per liter, gave precipitates with iso-amyl, heptyl and benzyl mercaptan. On buffering the solutions with sodium acetate, the intensity and the rate of precipitation, excepting perhaps with heptyl mercaptan, varied directly as the concentration of the buffer. The mercaptans were all added in excess. The precipitates produced by heptyl mercaptan were more intense than those with iso-amyl mercaptan.

Equal amounts of benzyl mercaptan were added to solutions of 1 mg. Co per liter, of varying alkalinity, in an atmosphere of N_2. After standing overnight, the test tubes had precipitates of either pink, or tan, or both colors. The same amount of benzyl mercaptan, when dissolved in alcohol and added to a buffered solution of Co, with a final concentration of 1 mg. Co per liter, produced a pink precipitate immediately. Additional alcohol dissolved this precipitate, leaving a pink solution. Evaporation of the alcohol reproduced the pink precipitate. An alcoholic solution, containing one-twentieth the amount of benzyl mercaptan used above, added to a Co solution of 1 mg. per liter concentration, had no effect. Repeating this with one-fifth the amount of mercaptan did produce a precipitate. Less than this amount of mercaptan had no effect.

N-amyl mercaptan in excess, added to buffered solutions containing 1 mg. of metal per liter, produced turbidity immediately with Cu, Pb, Co and Sb, but with Ni, Ag and Fe only on standing. After one hour the Ni solution had a greenish tint, the Co solution was pink and the Zn solution had the appearance of a precipitate, though hardly turbid. A stoichiometric amount of the mercaptan in alcohol had no effect on solutions of Cu, Ni, and Co of the above concentration.

N-propyl mercaptan in excess produced a pink solution with 1 mg. Co per liter and a green solution with 1 mg. Ni per liter. N-propyl mercaptan seems to be more soluble in water than the n-amyl mercaptan.

A saturated aqueous solution of benzyl mercaptan containing 5 per cent sodium acetate, showed a precipitate with Pb at a concentration of 1.3 mg. per liter. A Cu solution of that concentration showed a Tyndall cone different from that in the blank, but of similar intensity to that in a solution of concentration of 20 mg. Cu per liter.

Diphenyl Thiocarbazid, $(C_6H_5.NH.NH)_2C{=}S$. This was tested against the ions of a number of heavy metals in solutions buffered alkaline with sodium acetate. The ionic concentration of metal ion was kept at 1 mg. per liter and the concentration of the thiocarbazid was well in excess of this. With none of the metal ions tested (Cu, Pb, Ni, Co, Sb, Zn, Ag, Fe) was there any visible precipitate on mixing solutions, although distinct Tyndall-cone effects were observable with Cu, Pb, Ni and Co. With time (the mixed solutions were observed at intervals over a period of six days), precipitates formed with all of the ions except Sb, but precipitation was earliest and most plentiful with Co and Ni. On the strength of this observation diphenyl thiocarbazid was found to be commercially useful for differential flotation of siegenite (Co, Ni sulfide) away from copper-bearing sulfides and pyrite.

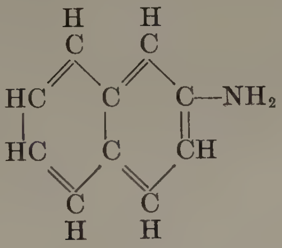
Thiocarbanilid has such a low solubility in water that it is difficult to work with aqueous solutions. An alcoholic solution added to alkaline buffered metal-salt solutions of 20 mg. per liter metal concentration gave a heavy brown precipitate with copper, but a white precipitate, indistinguishable from the precipitate of the compound itself, when alcoholic solutions were added to water, with Co, Ni, Pb and Zn.

Sodium Diethyl Di-thio Carbamate $(C_2H_5)N.CS.SNa$.—An excess of this salt in aqueous solution forms a yellow-brown precipitate with Cu (40 mg. per liter) solutions, both unbuffered and alkaline buffered with sodium acetate; with Pb (40 mg. per liter) the precipitate is white, much denser in the alkaline buffered solution; with Co (40 mg. per liter) a yellowish green turbidity forms in the unbuffered solution and a flocculent precipitate of the same color in the buffered solution; Ni (40 mg. per liter) gives a yellow flocculent precipitate in both buffered and unbuffered solution; the Zn (40 mg. per liter) precipitate is white, heavier in the buffered solution; Ag (40 mg. per liter) gives a white turbidity only; Fe (40 mg. per liter) a dense muddy brown precipitate both buffered and unbuffered.

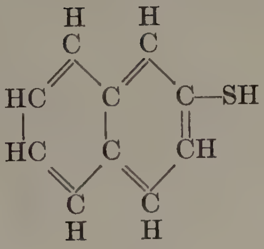
It is interesting to attempt to follow the adsorptionist reasoning as to results like these. The simple straightforward interpretation would seem to be that when heavy-metal ions and ions or molecules with a reasonably labile acidic hydrogen atom, of the organic types tested, are mixed in water, metathesis occurs. Even the most rabid adsorptionist could hardly deny this conclusion, if he considered these experiments alone. But when the metal ions are not in solution but form a part of the crystal lattice of a mineral particle in a flotation operation, the adsorptionist abandons the simple and direct analogy for the abracadabra "adsorption." He would apparently rather, if he thinks of it at all, place his reliance on an assumption of surface-tension changes, at an interface (liquid-solid) at which no definite determination of even the existence of a tension, let alone its magnitude, has ever been made, than to believe that an ion anchored in a solid lattice can react ionically in the same way that it can react if free-swimming in solution. And this in the face of the fact that reaction products, involving such reactions, are recoverable from the solid surfaces. He will assert that this adsorption of his takes place only, or with the greatest facility, when reaction between an ion of the solid and ions or molecules of the adsorbate is possible. But he seems unable to conceive that that which is possible may possibly occur.

Nitrogenous Collectors

All of the collectors thus far considered have been of the acidic type, and have been shown capable, either specifically or as members of a class, of reacting metathetically with heavy-metal ions to form insoluble precipitates. There exists, however, a group of collectors, of which naphthylamine, diazo-amino-benzene, and thio-naphthol are representative, which have no recognized acidic tendencies, and two of which are, if anything, of basic character. Yet apparently they coat sulfide mineral particles from solution, and their hydrocarbon parts are apparently oriented as in the case of their acidic analogs. Thus the contact angles of β-naphthylamine



and of thio-β-naphthol



were found to be as follows:

Mineral	Contact Angle, Deg.	
	β-Naphthylamine	Thio-β-Naphthol
Galena	65	66
Pyrite	56	66
Chalcocite	55	66
Covellite	66	66

It was necessary to condition the galena and pyrite in the naphthylamine for 90 min. and the covellite for 15 min.; the chalcocite angle given is for 3 min. conditioning only. Five minutes conditioning only was allowed for galena, chalcocite and covellite in the thio-β-naphthol; one hour was necessary for the pyrite.

While it has been shown that the nitrogenous collectors (specifically α-naphthylamine and diazo-amino-benzene) are abstracted from solution by sulfides, no data pointing directly to chemical reaction as the mechanism of extraction exists. It was reasoned in a previous paper[35] that the adherent effect resided in the nitrogen group in the molecule. Evidence —not proof—that this action is chemical lies in the fact that chemical reactions between such groups and ions present in ore minerals to produce water-insoluble compounds are known. Thus p-nitrobenzene azoresorcinol forms precipitates with Mg, Ni and Co in strongly alkaline solutions;[36] α-hydroxyquinoline[37] forms a precipitate with Al.[38] This same reagent also precipitates magnesium[39] and zinc.[40] Benzidine precipitates Fe and Pt,[41] tungsten,[42] and copper.[43] α-Benzil dioxime,[44] dicyandiamidine sulfate,[45] and dimethyl glyoxime[46] precipitate Ni in alcoholic solution and the latter also in strongly alkaline aqueous solutions. p-Dimethylamino-benzal-rhodanine causes precipitates in dilute solutions of Ag, Cu and Hg;[47] also of Pd, Pt and Ir.[48] Cupferron precipitates both Fe and Cu from acid solutions.[49] Ethylene-diamine forms a precipitate with Cd.[50] These examples, chosen more or less at random from the Eastman list of organic chemicals useful in analytical work, is conclusive on the score of the possibility of reaction between ions present in ore minerals and nitrogenous collectors and, taken in connection with the proved behavior of acidic collectors, seems highly persuasive to the conclusion that the behavior of the nitrogenous collectors is similar.

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[35] Taggart, Taylor and Ince: Reference of footnote 20.
[36] Ruigh: *Jnl.* Amer. Chem. Soc. (1929) **51**, 1456.
[37] Kolthoff and Sandell: *Jnl.* Amer. Chem. Soc. (1928) **50**, 1900.
[38] Atack: *Jnl.* Soc. Chem. Ind. (1915) **34**, 936.
[39] Hahn and Vieweg: *Ztsch. anal. Chem.* (1922) **71**.
[40] Berg: *Ztsch. anal. Chem.* (1922) **71**, 171.
[41] Malatesta and di Nola: *B. Chem. Farm.*, **52**, 461.
[42] Knorre: *Ber.* (1905) **38**, 783.
[43] Spacu: *Ztsch. anal. Chem.*, **67**, 31.
[44] Atack: *Analyst* (1913) **38**, 316.
[45] Grossman and Schuck: *Chem. Zeit.* (1907) **31**, 335, 911.
[46] Weeldenburg: *Chem. Weekblad* (1924) **21**, 358.
[47] Kolthoff: *Jnl.* Amer. Chem. Soc. (1930) **52**, 2222.
[48] Feigl, Krumholz and Rajmann: *Microchem.* **3**, 165.
[49] Baudisch and King: *Jnl. Ind. & Eng., Chem.* **3**, 629.
[50] Spacu and Sucin: *Ztsch. anal. Chem.*, **77**, 340.

FLOCCULATION

It was set forth in the discussion of the paper that first stated the broad generalization of the chemical theory,[51] that not chemistry but "flocculation" was the thing to study if we would arrive at a practical understanding of the flotation process. This was, apparently, the critic's understanding of the millman's slogan that "to float a mineral you must first flocculate it." The point of view is ably set forth by Sulman[52] and adopted by Holman.[53] Gaudin and Malozemoff[54] put forth a similar postulate to explain the poor flotation of a part of the very finely ground mineral in flotation pulps.

Much of the confusion of thought that exists on this subject is due, the authors believe, to a lack of definition of terms, based on failure to observe closely. The millman's creed originated in the days of oil-froth flotation, and was substantially equivalent to saying that to float mineral you must first form it into a froth. The great majority of the mineral floccules observable during and after an oil-flotation operation are air-bound; i.e., they are aggregates of air bubbles and minerals, insufficiently aerated to float. Such floccules are characteristic of all of the pulp-body flotation processes.[55] But floccules of such size, indeed of any size sufficient to be observable with the naked eye or with a low-power hand glass, are substantially absent in modern flotation with soluble collectors and almost completely dissolved frothers (e.g., cresylic acid or pine oil in quantities of 0.1 lb. per ton of ore or less).

On the other hand, flocculation of the fine mineral into non-air-bound aggregates too small to be resolved by the naked eye or by a low-power glass does occur in properly conditioned modern flotation pulps and, occurring, is evidence of the completion of the desired chemical reaction at the surfaces of these particles.

It was pointed out in an earlier paper[56] that the behavior of the minerals quartz, galena and sphalerite in a differential flotation operation paralleled their behaviors with respect to Brownian movement and dispersion (suspension) in the presence of the various flotation reagents used. The observations there set down have been verified many times each year by students in the regular flotation classes at Columbia. It was further postulated that this parallelism followed from the chemical state of the particle surfaces and that Brownian movement and high dispersion denoted a surface ionized and therefore water-wetted, while lack of Brownian movement, accompanied ordinarily by flocculation, signified either an insoluble unionized surface, or one so highly soluble that if ionization of the dissolved surface material existed, there was insufficient pull between the ions of the dissolved portion and the ions of the lattice to anchor one of the former. Much work has been done on this postulate since it was announced. A part of the results is already published;[57] the remainder is doctorate research[58] as yet unpublished. It demontrates, however, that in suspensions of zinc sulfide in disodium arsenate, maximum dispersion and Brownian movement occur within a relatively narrow concentration band, on either side of which the zinc sulfide particles are flocculated and at rest; that within this dispersion range the zinc sulfide particles are coated with zinc arsenate, and negatively charged; that the arsenate ions are anchored to the surface of the particles and move with them, under the influence of an imposed field, to the positive electrode, while the zinc ions move, at the same time, to the negative pole. Both ions are discharged at the electrodes and the discharged sulfide particles flocculate and sink. Further, it has been shown that at arsenate concentrations below the critical, the failure of Brownian movement is due to insufficient surface coatings of zinc arsenate, while at concentrations above the critical the excess of sodium arsenate dissolves zinc arsenate to form a double salt, thereby leaching zinc arsenate from the particle surfaces as fast as it forms.

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[51] Taggart, Taylor and Knoll: *Trans.* A.I.M.E. (1930) **87**, 217.

[52] Sulman: Inst. Min. and Met. (1930) *Bull.* 311.

[53] Holman: Inst. Min. and Met. (1930) *Bull.* 314.

[54] Gaudin and Malozemoff: page 303 this volume.

[55] Taggart: Handbook of Ore Dressing, 790. New York and London, 1927. John Wiley & Sons and Chapman & Hall.

[56] Reference of footnote 51.

It appears, therefore, that flocculation in and of itself is neither a prerequisite to flotation nor an indication of readiness to float. But rather it and its converse are indications of the surface chemical condition of the pulp particles. We may say with assurance that a particle of a given mineral in Brownian movement will not float, because it is water-wet, and further that the surfaces of particles of the same mineral in the same pulp, not in Brownian movement because too large, are in the same water-wet state and that the particles cannot, therefore, float. But the converse will not be true unless the failure to disperse and the concomitant tendency to flocculate are brought about by coating the particles with an insoluble water-repellent film. Under such circumstances flotation will be due to the film and not to the flocculation. The flocculation is merely one of the indicators of the existence of such a film.

The experiment cited by Gaudin and Malozemoff to the effect that "near-colloidal" galena previously dispersed by inorganic agents and then added to quartz could not be floated, whereas galena previously flocculated by other inorganic reagents and added to similar quartz could be floated, is not in conflict with this conclusion. On the contrary, it is readily explicable in terms of the hypothesis. The dispersed galena had an active ionized surface. On admixture with "near-colloidal" silica, which also has a similar active ionized surface (being in vigorous Brownian movement in distilled water) reaction is to be expected between the anchored ions at the respective particle surfaces (Pb^{++} at the galena surface and $SiO_3^{=}$ or another silicate ion at the quartz surface) to form a relatively insoluble lead silicate cement binding a quartz slime-coating to the galena surface and thereby changing it effectively to a gangue surface in so far as flotation goes.[59] The flocculated galena, on the other hand, had a relatively un-ionized and correspondingly inactive surface, which protected and preserved it against the slime-coating reaction, although it could be and was broken into by the collecting agent. In this connection del Giudice has shown that galena particles coated with lead silicate may be subsequently coated with lead ethyl xanthate with displacement of silica ion, but that, although lead ethyl xanthate is less soluble than lead carbonate and will xanthate-coat galena precoated with lead carbonate, or will condition galena against slime-coating with calcite slime, it will not displace a calcite slime-coating already formed from the surface of a galena particle. Thus the Gaudin and Malozemoff result is not an evidence of the necessity for flocculation *qua* flocculation prior to the flotation even of "near-colloidal" material, but rather cumulative factual support of the chemical hypothesis.

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[57] Taggart: *Jnl. Phys. Chem.* (1932) **36**, 130–153.

[58] T. C. Fitt: Columbia University, 1934.

INHERENT FLOATABILITY OF MINERALS

It may seem odd, at this date, to resurrect so old a friend as the inherent floatability of minerals, and would be so had not a recent writer[60] unearthed the ancient fossil for us and dressed it up in modern-appearing clothes. Gaudin[61] gives a table in which the most easily floated minerals of metallic luster are arranged in a rough order of ease of flotation, but he prefaces his list with a statement of cause that exonerates him from support of any theory of inherency. Sulman,[62] on the other hand, as late as 1930, restates conclusions published in 1919[63] based on experimental work running back to 1907, and gives a list of substances arranged in order of their floatability in pure water. Patek, adapting the bubble-machine method of estimating floatability, amplified the list and, in the opinion of the authors, perpetuates the error.

The fact is that, if contact angle is accepted as a reliable means of estimating floatability,[64] then the inherent floatability of Sulman and

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[59] del Giudice: See page 398, this volume.

[60] Patek: See page 486 this volume.

[61] Gaudin: Reference of footnote 5, 38.

[62] Sulman: Inst. Min. and Met. *Bull.* 311(1930).

[63] Sulman: *Trans.* Inst. Min. and Met. (1919) **29**, 44.

[64] The authors are convinced that it is. For argument *contra* see Gaudin: page 233, this volume.

Patek, meaning resistance to water wetting in the presence of air, is a myth. For the reason that water wets clean mineral surfaces in the presence of air completely, there is not contact angle. This fact has been suspected by one of the authors for many years,[65] but it was first demonstrated experimentally by Wark and Cox and has since been verified exhaustively in the Columbia laboratory. It may now be stated, without fear of contradictory showing, that air will not displace water from a clean surface of any one of a large majority of the minerals, both metallic and nonmetallic, met with in flotation.[66]

The discrepancy in experimental results as between different investigators, with consequent disagreement in conclusion, follows from the difficulty of obtaining clean surfaces for test. If a surface is prepared by grinding and polishing under water, on a grease-free lap, using abrasive free of oil and grease or of dissolved organic materials of the collector class, holding the test specimen in such fashion as to eliminate the possibility of contamination with grease from the fingers, the resulting polished surface will remain evenly covered with water when removed therefrom, and a captive air bubble will not contact with it. If the particle is greasy, that fact will be evidenced by drawing together of water into droplets on a surface withdrawn from water, and by contact in the bubble machine. Such accidental contamination can always be removed and further contamination eliminated by sufficient care in the preparation of specimens.

A clean surface will not remain clean, however, if it is permitted to dry in air. Invariably such an air-dried particle fails to become completely wetted when immersed in clean running water, and just as invariably it shows a contact angle in the bubble machine. This is where the measurements of both Sulman and Patek fall down. Sulman's measurements were made on selected crystal faces of dry minerals which were then partly immersed in the upper surface of a body of water and the contact angle measured. The specimens were attempted to be cleaned by successive washings in ether, alcohol and distilled water.[67] It is only necessary to try this method on a surface known to be contaminated in order to be convinced of its ineffectiveness. Hence Sulman's test particles, which had been freely handled and exposed to the air before cleaning, and were ineffectively cleaned, may be taken to have been contaminated in all cases, and his measurements to have no meaning other than, perhaps, a slight indication of relative adhesion of grease films to different minerals. Even this indication is predicated upon a most unlikely assumption of initially equal degrees of accidental contamination.

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[65] Reference of footnote 35, 295.

[66] The limitation in the statement of this sentence to less than all minerals is inserted solely on account of the fact that experimentation has not embraced all minerals. So far as experimental experience goes, no exception to the rule has been encountered.

[67] H. L. Sulman and H. K. Picard: Concentration Processes Involving Surface Tension. Unpublished brochure, about 1907, produced as an exhibit in Proceeding No. 331 in Equity in the District Court of the U. S., District of Delaware, Minerals Separation, Ltd., *vs.* Miami Copper Co.

Patek ground his test specimens wet, then treated them successively with hydrochloric acid, distilled water and ether, permitting drying between distilled-water washings. Our experience, as has been previously stated, is that water-washing followed by air-drying results in contamination invariably, and that further washing does not remove the contaminant film. In attempting to verify Patek's results, using clean, wet-polished mineral surfaces, we found that the dry contact angle[68] with water varied according to the day of the week, the time consumed in drying, and the method of handling the test specimen; i.e., whether with fingers, forceps, etc. In no case did an air-dried particle that before drying had given a wet contact angle[69] of zero in clean water give a zero wet contact angle thereafter. The wet contact angles on such materials were rarely reproducible with any degree of exactness, but they ranged, in general, between 35° and 50°. The latter angle is typical of a heavy contamination with body greases and of fatty acids of molecular weight in the neighborhood of that of oleic acid.

Consequently, we dissent vigorously and finally from any idea of inherent natural floatability. We admit that certain minerals are more readily soluble than others or are more readily oxidized and thereby rendered surface-soluble and ready to react with collectors, but beyond this we do not go, nor do we recognize any persuasive evidence pointing in such a direction.

CONDITIONING

It is interesting to note that although the quantitative evidence of chemical reaction between conditioning reagents and the minerals in flotation pulps is by no means so complete or convincing as that available with respect to the acidic collectors, the acceptance of chemical hypotheses to explain the conditioning action is much more ready. This is perhaps due to the fact that most conditioning agents are inorganic and that it is easier for the miner and metallurgist, trained in inorganic but not organic chemistry, to think of the behavior of such inorganic compounds in the familiar terms of ionic metathesis, rather than in the less familiar terms of surface tension and adsorption, while he lumps all his unfamiliarities together when organic collectors enter the picture.

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[68] Liquid drop in air.

[69] Captive air bubble in liquid.

Activation.—Gates and Jacobsen,[70] McLachlan,[71] and Gaudin[72] unquestioningly accept the statements of the geochemists[73] that conditioning of sphalerite by copper sulfate is simple metathesis resulting in a surface coat of covellite on the sphalerite particle. Abstraction of copper ions from copper sulfate solutions by powdered sphalerite, and simultaneous throwing of zinc ion into solution are readily observed. Sulfate-ion concentration in the solution remains substantially constant. Replacement of zinc in the relatively soluble zinc sulfide by copper to form a much less soluble copper sulfide is, therefore, the indicated reaction. But whether this sulfide is covellite or not has not been established by any quantitative work dealing with flotation pulps. In fact, such evidence as is available on this score might be considered *contra.* Thus Wark and Cox[74] find that the cyanide effect on copper-activated sphalerite is different from that on massive covellite, and in a later publication[75] the same authors state that activated sphalerite and chalcopyrite act in the same way toward both collectors and activators, although the sphalerite is somewhat more sensitive to the action of depressors. If the ordinary formula for chalcopyrite, $Cu_2S.Fe_2S_3$, is accepted, we have here from the adsorptionist standpoint a rather amusing conflict of evidence, since it is difficult to explain why a surface coating of divalent-copper sulfide should adsorb like a massive sulfide containing monovalent copper rather than like the massive sulfide of divalent copper. From the standpoint of the chemical theory, however, the conflict is unimportant. With both chalcopyrite and covellite the soluble oxidation product of the copper is cupric sulfate and the reaction product with the collector will be the same. In this case the relatively greater resistance of massive covellite than of activated sphalerite toward cyanide, and the relatively equal resistances of chalcopyrite and the activated sphalerite toward the same reagent, are in line with the fact, also developed by Wark and Cox, that in the series of copper minerals resistance to cyanide increases with copper content. Whether this means that high copper content in the original mineral results in correspondingly high copper concentration in the ore pulp, and consequent high consumption of cyanide ion by tying it up in cupricyanide ion—which seems reasonable—or whether some other explanation is the true one, is not known. The fact remains that interchange occurs between zinc and copper ions, that the copper ions go with the solid sphalerite and must, therefore, be anchored to it, presumably in association with the same ion that originally held the displaced zinc, and that the altered surface will remove collector ions from solution.

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[70] Gates and Jacobsen: Utah Eng. Expt. Sta. *Bull.* 16 (1925).

[71] McLachlan: Canadian Inst. Min. and Met. (1926) **19**, 987.

[72] Gaudin: Reference of footnote 5, 76.

[73] Emmons: U. S. Geol. Survey *Bull.* 625 (1917) 140.
Watanabe: *Econ. Geol.* (1924) **19**, 497.

[74] Wark and Cox: Page 245, this volume.

[75] Wark and Cox: Page 267, this volume.

It is assumed quite freely by Gaudin and his coworkers, and the present authors are in agreement, that the activating action of Hg, Ag and Pb salts is due to similar metathesis resulting in similarly altered coatings on the minerals that they activate. But direct evidence for such assumption is scanty.

Gaudin and others[76] assign the activation by copper and lead salts of calcite floated with soap to exchange of Ca with Cu and Pb ions at the calcite particle surfaces. They assert that the copper and lead soaps are less soluble than the calcium soaps. No evidence is adduced on this score, but it is not out of line with the general behavior of metallic soaps. As to the facts of ion interchange, however, confirmation lies in a Columbia experiment in which green crystals and greenish coatings were observed on a microscope slide of copper-conditioned calcite, and these crystals and coatings, as well as surfaces not visibly discolored by the copper treatment, were turned brown to black when H_2S was introduced. Similar blackening occurred with lead-conditioned calcite, although no change had been visible (probably because lead carbonate is white) before the addition of H_2S.

There would seem, therefore, to be no great scientific recklessness involved in accepting the postulate that the mechanism of activation by heavy-metal salts involves metathesis between the activator and a surface ingredient of the mineral, resulting in deposition of a compound of the metal of the activator at the mineral surface. We may go even further and say that, with a complete and reliable table of water solubilities at hand, a suitable activator can almost certainly be picked before trial as a salt, the cation of which forms with the anion of the mineral to be floated a less soluble compound than the mineral itself, and with the anion of the collector a less soluble compound than that formed between this same ion and the cation of the mineral. The one further limitation is that the reaction product between activator and mineral must not be both highly insoluble and highly resistant to chemical reaction. Thus, if the copper sulfide coating on sphalerite were resistant to oxidation, the sphalerite could not be floated by ethyl xanthate, for the reason that the copper sulfides are less soluble, in the absence of oxygen, than the copper xanthates. But since copper sulfide, especially when first precipitated, oxidizes in the presence of water with great readiness, and the oxidation products are soluble in water, the copper ion is thus made available for reaction.

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[76] Gaudin: Univ. Utah *Tech. Pub.* 1 (1928).

Ravitz and Porter[77] cite, as one of their arguments, against the chemical theory, the fact that what they call "pure gold" floats, and that Wark and Wark[78] obtained the usual ethyl xanthate contact angle on gold surfaces. This argument is interesting here for the twofold reason that in the first place, far from being an argument against the chemical theory of collector action, it is entirely in line with that theory; and in the second place, because it brings up an interesting and highly practical example of an activation operation involving a mechanism different from that met in activation by heavy-metal salts.

Considering first the "pure gold" part of the Ravitz and Porter position, it is certainly safe to assert that pure native gold is never floated. Clarke[79] says that the purest native gold yet found contained 0.2 per cent of impurity, principally copper, and he gives analyses of samples from all over the world, ranging in gold content from 39 to 93 per cent, and carrying as alloy metals silver, copper, iron, mercury, palladium, rhodium, bismuth and tellurium. Gold flotation with xanthates involves, therefore, the collection of an alloy, almost invariably containing silver and usually copper, both of which metals oxidize readily in water and just as readily form relatively insoluble xanthates.

As to the matter of contact angles on pure gold, we are inclined to disagree with the Wark and Wark results, or at least with the Ravitz and Porter interpretation of them. Using gold of the highest purity that we were able to obtain, we could not get air-bubble contact in ethyl xanthate solution without pre-activation. Using ordinary commercial gold, contact was readily obtained. Such commercial gold, however, gave strong tests for both silver and copper. Prepared alloys of our refined gold with copper and with silver also gave contact angles readily with ethyl xanthate. We, therefore, believe that Wark and Wark, having no particular reason to inquire too closely into the purity of their gold specimen, used one of the usual accidental alloys.

Our "pure gold," however, could be activated by pretreatment in either nitrohydrochloric acid (*aqua regia*) or alkaline cyanide solution. Since both of these reagents react with gold, forming the chloride and a cyanide of the metal respectively, and since these compounds are both soluble in water and their solutions form precipitates on admixture with aqueous ethyl xanthate solutions, the weight of the evidence would seem to be that their activating function consists in rendering the gold at the surface of the gold particles into soluble form, ionic form, thereby promoting metathesis with xanthate or other suitable similar collectors.

A similar type of activation is probably involved in conditioning cassiterite with sodium hydroxide in order to effect flotation, using cupferron as a collector. The tin salt of nitrosophenyl hydroxylamine (tin derivative of cupferron) is sufficiently insoluble in water, and the hydrocarbon group sufficiently water repellent to constitute cupferron a suitable collector for tin minerals, if the tin ion of the mineral can be freed. Sodium hydroxide reacts with tin oxide in such a way as to do this.

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[77] Reference of footnote 12.

[78] Wark and Wark: *Jnl. Phys. Chem.*, (1933) **37,** 805.

[79] Clarke: The Data of Geochemistry. U.S. Geol. Survey Bull. 330.

It is difficult to imagine an adsorption hypothesis for these activation operations, and, since none has yet been set up, we must leave refutation to the future, in the hope that further advance in our factual knowledge will make it then unnecessary.

It is a question whether the use of xanthates with tar oils in gold flotation and of soaps with fuel oils in the flotation of certain nonmetallic minerals does not constitute a third type of activation rather than a collecting operation. Both in nonmetallic flotation and in those gold-milling operations in which the use of tar oil is necessary, although some collection is possible without the oil, satisfactorily complete recovery cannot be thus obtained. In the bubble machine, air-bubble contact is correspondingly erratic and tenuous. Without the xanthate or the soap, respectively, but with the oil, flotation is highly unsatisfactory, air-bubble contact is correspondingly weak or impossible, and captive oil bubbles make weak contact with gold and doubtful or no contact with the nonmetallics, depending on the oil used. But in the presence of the so-called soluble collectors (the xanthate and soap) a captive oil bubble makes ready contact, irrespective of the oil used, and a particle thus doubly conditioned (i.e., with soluble collector and oil) makes ready and strong contact with an air bubble.

Christensen[80] undoubtedly had the right idea concerning this phenomenon when he pointed out that acidic organic compounds, e.g., fatty and resin acids, reacted with certain nonmetallic minerals to coat them with insoluble soaps, and that thereafter these coated particles would behave toward oil as sulfides behave, becoming oil-filmed and water-repellent. We now know that the sulfides must have their reaction coating before they will oil-film, but that does not detract from either the reliability or the brilliancy of Christensen's observation.

Activation of metallic oxides by means of soluble sulfides is clearly chemical. It involves formation of the corresponding sulfide and is done for the twofold purpose of decreasing the consumption of collector and producing an oriented film of the reaction product of the collector anion and the mineral cation.[81] The use of soluble sulfides is not highly effective, however, on account of the fact that an excess of sulfide is desirable in order to insure complete filming, but at the same time harmful because it prevents the oxidation necessary before the collecting reaction can occur. It is probably to obviate this difficulty, although unrecognized, that sodium trithiocarbonate has been suggested for activating oxidized metallic ores. This compound decomposes slowly to yield sulfide ion, thereby tending to prevent excessive consumption by deep-seated reaction with the oxide, while at the same time the concentration of sulfide ion is held relatively low and reoxidation to the extent necessary for collection is not prevented.

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[80] Christensen: Reference of footnote 2.

[81] See discussion of cerussite, *Trans.* A.I.M.E. (1930) **87**, 247 and 357.

The use of ammonium phosphate in oxidized lead flotation[82] is another example of the same type of activation, lead phosphate being much less soluble than many of the lead compounds in an oxidized ore, but more soluble than lead ethyl xanthate.

Depressors.—The most extensive research dealing with this class of reagents is the work of Wark and Cox[83] on cyanide and copper sulfate. The work is largely on the effect of changing cyanide-ion concentration on the contact angles obtained with various acidic-sulfur collectors, and contains very little of quantitative chemical nature. For this reason the argument therefrom as to the chemical hypothesis is largely inferential, and gains such weight as it may carry from the completeness with which the observed phenomena are explicable in terms of known chemical reactions and generally accepted chemical "laws."

Wark and Cox find that cyanide, in suitable concentration, depresses sphalerite that has already been activated with copper sulfate, using a variety of acidic-sulfur collectors; that it prevents activation of sphalerite by copper sulfate unless the copper is present in a proportion exceeding one Cu to three CN; that the quantity of alkaline cyanide necessary to effect depression varies with the pH of the solution, but that the relation is such that the free cyanide ion at the critical point (between contact and no-contact) is a constant quantity; and that the curve of constant cyanide-ion concentration, and of bubble contact, is also a precipitation curve for the copper salt of the collector (specifically cuprous ethyl xanthate). The authors postulate that the cyanide may act in any one, presumably more, of the following three ways, to dissolve, or prevent formation of: (1) the activating copper-bearing film; (2) "a film consisting of xanthate adsorbed by the activating film"; (3) the xanthate film without the activating film.

The weight of the evidence seems to point to the correctness of postulate 1 when cyanide treatment precedes treatment with collector, since the transition point between contact and non-contact coincides with a Cu:CN ratio of 1:3, with contact when the copper exceeds this ratio and vice versa. The action with preactivated sphalerite may be either solution of the precipitated copper sulfide, which readily occurs, or immediate reaction with and withdrawal of copper ion made available by oxidation. The correctness of this hypothesis is strengthened and—as the authors point out—the incorrectness of postulate 3 is indicated by the fact that a sphalerite particle that failed of contact in a xanthate-cyanide-copper sulfate solution containing cyanide in excess of the 3:1 ratio, failed to respond to xanthate after removal from the original solution and subsequent washing. On the other hand, when the collector is present during conditioning, the amount of cyanide required to depress varies with the collector. Hence postulate 1 cannot be the whole truth.

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[82] Christmann and Falconer: *Eng. and Min. Jnl.* (1929) **127,** 951.

[83] Wark and Cox: Pages 189, 245 this volume.

The second postulate is in language that leaves the meaning entirely uncertain. It may refer to a film of copper sulfide plus "adsorbed" molecular potassium xanthate, which we think impossible for reasons already pointed out; or it may mean copper sulfide plus "adsorbed" xanthate ion, which we likewise disagree with as a possibility (see p. 356). But in either case, since cyanide will dissolve copper sulfide formed by precipitating cupric ion with sulfide ion, the sulfide film would be dissolved away. Such accord with the adsorption postulate, however, as may be inferred from the fact that cyanide would dissolve these sulfide films, is exploded by the fact that xanthate will not cause bubble contact with a sphalerite surface coated with copper sulfide; i.e., activated with copper sulfate and then placed in and tested in a solution containing excess of sulfide ion. In this respect it is like galena and sulfide-activated cerussite.[84] Molecular or ionic "adsorption" theory must, therefore, imply adsorption on to an oxidized copper surface, presumably a highly soluble copper sulfate surface, and this is distinctly not in accord with the experimental background of colloidal chemistry.

If, however, the Wark and Cox postulate 2 refers to a film of cuprous xanthate, and the word "adsorbed" is mere surplusage, our experience is in full agreement, and the hypothesis accords well with their own experimental results. For precipitated cuprous xanthate dissolves readily in alkaline cyanide solution, and the precipitation curves cited by Wark and Cox show that the same compound will not precipitate in the presence of an excess of cyanide.

In their latest paper Wark and Cox say:[85]

> It now appears that the cyanide prevents adsorption of the xanthate film without otherwise changing the surface, except in the case of sphalerite previously activated by copper sulfate.

Presumably, in the excepted case they attribute the cyanide effect to solution of the activating film. The experiment cited in support of this postulate is that a curve of benzene contact-angle change with chalcopyrite in the presence of variable cyanide ion roughly coincides with the ethyl xanthate contact curve, also using cyanide as the depressant.

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[84] See *Trans.* A.I.M.E. (1930) **87**, 217 or 285.

[85] Wark and Cox: Page 267, this volume.

Just why this experiment, if verified, had the significance attributed to it is difficult to see in view of the fact that the contact curve with other collectors does not coincide with that for ethyl xanthate. But entirely apart from this, we find that carefully purified benzene, when used with every precaution against contamination, is like Nujol in that it does not make contact either with chalcopyrite or pyrite, even without cyanide present. The presence of a most minute amount of fatty acidic contaminant, as is introduced, for example, by touching the finger to the benzene, brings about contact. Thus, instead of constituting evidence for "adsorption," this experiment is explicable only on the basis of the chemical hypothesis, the fact being that the mineral surface must first be conditioned with a chemical agent with which it can—and inferentially does—react, before it will make contact even with a hydrocarbon.

The action of cyanide on sphalerite with collectors such as the higher dithiocarbamates and xanthates, which apparently require no activation,[86] probably involves a solubility balance between zinc-cyanide complexes, zinc sulfide and the zinc salt of the collector anion. The fact that cyanide causes dispersion and Brownian movement of sphalerite[87] is evidence, persuasive to the authors, of a relatively slightly soluble surface coating, not zinc sulfide, since dispersion of sphalerite does not occur in distilled water. Since sphalerite abstracts cyanide ion from solution, the coating is probably a zinc cyanide. Being sufficiently soluble to effect Brownian movement, this surface is replaced by the more insoluble zinc salt of the collector anion.

The facts pointed out by Wark and Cox[88] that the depressing effect of cyanide per unit of concentration, using ethyl xanthate as the collector, decreases through the mineral series pyrite, chalcopyrite, bornite, covellite, and chalcocite; and that the contact curve for pyrite with copper sulfate and cyanide is a curve of constant cupricyanide-ion concentration, taken together, seem to point to an iron-copper-cyanide complex as a possibly important factor in the depressing phenomenon throughout the series. The iron-copper ratio in the minerals decreases from pyrite to chalcocite (bearing in mind that it is extremely difficult to obtain either copper-free pyrite or iron-free chalcocite and covellite). Precipitates are produced at very low concentrations of salts introducing ferrous, ferric, cupric and cyanide ions, the color of the precipitate varying according to which of the various ions are present and their relative concentrations.

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[86] Wark and Cox: Reference of footnote 85.

[87] See *Trans.* A.I.M.E. (1930) **87**, 217 and p. 367, this paper.

[88] Wark and Cox: Page 258, this volume.

Significantly, the higher the ratio of copper to iron, the greater the quantity of cyanide ion required to produce a precipitate in a solution carrying these three ions. This points to surface coating, but whether the coating is cuprocyanide or cupricyanide, ferrocyanide or ferricyanide, or whether it is the same throughout the series, cannot be said.

A less familiar example of use of a depressant is that of sodium dichromate to drop lead away from copper sulfides. Usual procedure is to treat a collective float. Dichromate oxidizes xanthates. It, therefore, decomposes the xanthate coating on the floated galena. It then reacts with lead ion to form lead chromate at the galena surface, to such an extent as to be readily visible if an excess is used, and, lead chromate being less soluble than lead ethyl xanthate, the chromate ion cannot be displaced subsequently by xanthate ion in solution. Hence the lead fails to float on refrothing.

Gangue Control.—The ignorance of investigators on this score is probably greater than in respect to any of the other reagent functions. We know that with the fatty and petroleum oils of early froth flotation the addition of acid to the pulp usually increased recovery and raised the grade of concentrate as compared to unmodified pulps, that with the creosotes and tar oils of the later days of the oily reagents neutral or slightly alkaline conditions were equally good or superior on the same ores, while with the adoption of the sulfacidic type of soluble collectors the requirement changed, in general, to that of a pH ranging from 8 to 10. But so far as the literature or the knowledge of the authors goes, the underlying reason for these requirements is almost completely unknown.

What few facts we have, however, point to the conclusion that the phenomena underlying gangue control are simple chemical reactions, as are those controlling elsewhere through the process. Thus, certainly, one of the ways, at least, in which recovery is increased by so-called gangue-control reagents is by preventing coating of the sulfide minerals by gangue during the pulp-preparation period, and this del Giudice[89] has shown definitely to be a phenomenon of metathesis.

Examination of a number of pulps has shown unmistakably that control of slime coating is almost invariably the explanation of improvement in recovery following the introduction of the right gangue-control reagent to the pulp. In all of the cases examined, the sulfide mineral particles in the tailing before the addition of the gangue-control reagent were more or less heavily slime-coated while those in the concentrate were more lightly so. After the addition of the reagent, slime coatings were substantially absent.

But discovery of the phenomenon and its explanation is only the beginning of its effective control. Thus the slime present in Anaconda ore coats galena, chalcocite, chalcopyrite, sphalerite and pyrite in a pulp made up of water and the organic collector and frother only, but not all of them are coated to the same extent. Reduction in coating can be effected by adding either sulfuric acid or lime, both reagents being required in considerable quantities. The unmodified pulp itself is rather strongly acid, and yields qualitative tests for almost every common cation and anion. The slime is clayey material of almost completely unknown chemical composition. Whether the part of the slime that adheres to the sulfides is the same in the case of all of the sulfides; whether the adhering slime is a representative cross-section of all of the slime; whether the dissolved salts play a part in the coating reaction and coating control; are all unanswered questions of quite apparent complexity. But we have the nature of the problem clearly defined and the method of attack indicated. Solution involves, therefore, merely application of sufficiently delicate analytical methods to determine the constitution of the coating material and thereafter informed control of the reactions of this material. Thus, having discovered that lead carbonate is the cementing material in the coating of galena by calcite, we are not limited to sodium silicate for prevention. Introduction of any ion that will react with either the calcite or the galena to form a surface on the respective particles less soluble than lead carbonate will do the trick. Potassium ethyl xanthate does it by closing up the galena surface, as does also sodium phosphate. Sufficiently insoluble salts of calcium are more difficult to find, but this difficulty will not necessarily crop up with another gangue mineral.

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[89] del Giudice: Page 398, this volume.

On the other hand the reagent must not close up the sulfide surface too effectively. Thus while both phosphate and silicate ions are displaced from a galena surface by ethyl xanthate ion, chromate ion, which also prevents calcite coating, is not displaced by ethyl xanthate ion, so galena "protected" by this ion cannot be floated by the xanthate.

pH Control.—Gaudin[90] et al. showed that for a number of soluble collectors maximum recovery was obtainable only in a given pH band, narrow with some and wide with others. The Wark and Cox work, especially the latest paper, gives the beginning of the explanation of Gaudin's observation by showing that there is a critical pH with each mineral and each collector above which air-bubble contact is impossible, and near which it is weak and tenuous. This accounts for the fall in recovery at the alkaline end of Gaudin's curves. Failure in the acid range is due, certainly in some cases and probably in most, to decomposition of the collector.

We are indebted to Wark and Cox for information also as to another effect of pH control in their showing that, in the use of cyanide depressors, the cyanide-ion concentration is thereby regulated.

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[90] Gaudin et al.: Univ. Utah. *Tech. Pub.* 7 (1930).

Another type of chemical reaction that apparently is an instance of pH control, although actually it involves another type of phenomenon entirely, is involved in the use of lime, sodium sulfide and the like in ores containing relatively high percentages of soluble metallic ions such as iron and copper. The controlling effect of copper ion in differential flotation of lead and zinc and in pyrite flotation has already been fully developed. It also will precipitate and render substantially unavailable a stoichiometric amount of collector anion. Ferric ion similarly removes collector and also, if we may draw on the experience of the hydrometallurgists and geochemists, enters into a variety of reactions with the other ore minerals. Ferrous ion, probably entirely apart from its function as a source of ferric ion, exerts a harmful effect in certain flotation pulps.[91] These and other ions are precipitated and rendered substantially harmless by both hydroxyl ion and sulfide ion, and substantial completion of the reaction is indicated in both cases by an alkaline pH.

CONCLUSION

Thus it would seem that the actions of collectors, both those of the soluble acidic type and the oils, are rationally explained in terms of accepted chemical tenets by the chemical hypothesis of flotation, and that, insofar as evidence is available for the nonacidic nitrogenous collectors, chemical reaction with the ore mineral is indicated. As to the accessory reagents, such dispute as there is relates rather to the nature of the chemical reaction involved than to the question whether chemical reaction occurs. For the benefit of those who have not worked actively in the field, it may be said that knowledge therein is growing rapidly, and that utilization of the chemical hypothesis to direct research is like the use of strong headlights on a mountain road, not every bump or crossing is brought into immediate view but essential glimpses of the road around the corner are afforded.

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[91] McLachlan: *Trans.* A.I.M.E. (1928) **79**, Flotation Practice, 235.

Action of Alkali Xanthates on Galena*

By T. Clinton Taylor† and A. F. Knoll‡

(New York Meeting, February, 1933)

Qualitatively, galena (native lead sulfide) reacts with aqueous solutions of the xanthates,[1] and has its surface sufficiently altered so that there is a tendency for air bubbles to attach themselves to the mineral crystals where there was no such effect before the treatment. This property is utilized in froth flotation operations where the mineral sulfide particles are raised into the froth and the gangue whose surface is not altered remains behind.

The results of a study of this reaction were reported[2] recently and certain tentative suggestions about the interaction of galena and the alkali xanthates were offered. In this paper, there is set down the results of a detailed quantitative investigation of the ions involved in this interaction with a view to finding out more about the condition existing immediately at the surface of a crystal like a heavy metal sulfide after treatment with an organic reagent.

Past experimental experience showed that finely ground galena shaken with aqueous potassium xanthate abstracts xanthate ion and at the same time sulfate ion, reducing ions of the type $(S_mO_n)^=$ where the ratio of m to n is less than 4:1 (as in sulfate), sometimes hydroxyl ion, are thrown into solution.[3]

Ions Involved in Xanthate-galena Interaction

The present quantitative study of the galena-aqueous potassium xanthate system shows that at the lower concentrations of potassium xanthate (less than about 25 mg. per liter) the amount of xanthate ion abstracted by the galena can be accounted for by the amount of reducing ions of the $(S_mO_n)^=$ type and sulfate ion thrown into solution. At these low concentrations of xanthate ion, hydroxyl ion is not in evidence. At higher concentrations of xanthate ion (greater than about 25 mg. per liter), the amount of xanthate ion abstracted cannot be accounted for by liberated sulfate ion and the reducing ions $(S_mO_n)^=$ together, and it is only at these higher xanthate-ion concentrations that hydroxyl ion can be found. That reducing ions of sulfur-oxygen type are associated with galena is evidenced by the fact that aqueous extracts of galena that had been exposed to air for a long time reduced iodine and permanganate solutions. A precipitate ($BaSO_4$) is also obtained if barium ion is added to the acidified aqueous extract. If precipitated $BaSO_4$ is filtered off and the filtrate oxidized with H_2O_2 more $BaSO_4$ is obtained. The aqueous extract also gives a white precipitate with $AgNO_3$, which rapidly turns brown and then black. This indicates thiosulfates or thionates.[4] The aqueous extract of freshly ground galena generally gives almost negative results when tested as described above, although, as will be shown later, detectable amounts of the above ions may ultimately be displaced from it.

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* The material reported here is taken from a dissertation presented by A. F. Knoll to the Faculty of Pure Science, Columbia University, in partial fulfillment of the requirements for the degree of Doctor of Philosophy. For details see original dissertation.

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[1] A. F. Taggart, T. C. Taylor and C. R. Ince: *Trans.* A. I. M. E. (1930) **87,** 285. Milling Methods.

[2] A. F. Taggart, T. C. Taylor and A. F. Knoll: *Trans.* A. I. M. E. (1930) **87,** 217. Milling Methods.

[3] Reference of footnote 2.

Experimental Procedure

The experimental procedure followed by the authors in the tests described in this paper was as follows:

Large pieces of galena,[5] (about 2-in. cubes) were broken down to pieces ⅛ to ¼ in. on edge. The pieces that had bright clean surfaces and contained no inclusions,[6] were ground by hand in a porcelain mortar and passed through a 200-mesh brass sieve.

Thirty grams of the powdered galena was put into a 500-c.c. Pyrex glass stoppered Erlenmeyer flask, containing 300 c.c. of aqueous potassium xanthate solution. The flask was then fastened to the periphery of a wheel 3 ft. in diameter and slowly revolved so that the powdered galena kept falling through the solution. Controls containing potassium xanthate solution alone were run also, and were carried through the same process (mixing, filtering, etc.) as the actual experiments. The galena was then filtered off on an asbestos mat[7] in a Gooch crucible.

The following determinations were attempted on aliquot portions of the filtrate:

(*a*) The total reducing capacity in terms of 0.002N iodine, which was the sum of that due to xanthate ion and the ions $(S_mO_n)^=$.

(*b*) The reducing capacity after the removal of xanthate ion as xanthic acid is due to the ions $(S_mO_n)^=$. The xanthate ion equivalency in terms of 0.002N iodine is given by the difference between determinations *a* and *b*.

(*c*) Sulfate ion.

(*d*) Hydroxyl ion.

(*e*) Sulfide ion (qualitative).

(*f*) Carbonate ion.

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[4] T. Takamatsu and W. Smith: Chem. Soc. (1880) **37**, 592.

[5] Galena obtained from the Ward Scientific Establishment, Rochester, N. Y. was marked as coming from Ottawa County, Oklahoma.

[6] The asbestos used showed no abstraction of xanthate when shaken with aqueous potassium xanthate. Filter paper is uncertain in its action. Some of it is acidic, and weakly alkaline solutions filtered through it become acid.

[7] Reference of footnote 6.

Because of the low concentrations worked with, it was found necessary to employ 0.002N iodine solution in the iodine titrations with starch as an indicator.

The volume of 0.002N iodine necessary to give a visible blue color with a starch suspension is appreciable. This factor must, therefore, be compensated for in the iodine titration. For example, 50 c.c. of water containing 2 c.c. of a 1 per cent potato starch paste required 0.75 c.c. of 0.002N iodine solution. The volume of iodine needed to give a visible blue color varied with the kind of starch used and with the ionic strength of the solution, and a blank correction is made to avoid error here.

It is possible to get a sharp end point with the minimum amount of iodine by adding for each 50 c.c. of solution to be titrated 5 c.c. of K_2SO_4. This salt affects the formation of the starch iodine complex.

The filtrates from the treatment of galena with high concentrations of alkali xanthate are alkaline ($pH = 9$). At this alkalinity iodine is changed to hypoiodite, iodate, etc., so it is necessary to bring the filtrate to a pH of 7.5 or 8 with 0.05M acetic acid (amount determined by a side titration with phenophthalein) before addition of the iodine. If the filtrate is made too acid ($pH = 5$ or less) xanthic acid is formed and decomposes to non-iodine consuming products.

Determination of Total Reducing Ions.—A 50-c.c. aliquot of the filtrate to which were added (after neutralization with 0.05 acetic acid) 5 c.c. of 0.2M K_2SO_4 and 2 c.c. of 1 per cent potato starch paste was titrated with 0.002N iodine. The titration was corrected by subtracting the value from a blank run as mentioned above.

The iodine titer is due to the interactions described by the following equations:

$$2(C{=}S.OC_2H_5S)^- + 2I^\circ \rightarrow \underset{\text{dixanthogen}}{(C{=}S.OC_2H_5S)_2} + 2I^-$$

$$(SO_3)^{=} + 2I^\circ + H_2O \rightarrow (SO_4)^{=} + 2I^- + 2H^+$$

$$2(S_2O_3)^{=} + 2I^\circ \rightarrow (S_4O_6)^{=} + 2I^-$$

The precision that could be obtained using 0.002N iodine was determined by means of potassium xanthate as a standard. On 50 c.c. of solution representing 1 to 10 mg. of potassium xanthate checks could be obtained within 0.3 per cent.

Determination of Reducing Ions Not Xanthate $(S_mO_n)^{=}$.—For the determination of reducing ions not xanthate, a 50-c.c. aliquot of the filtrate was acidified with 50 c.c. of 0.3M tartaric acid. The xanthic acid thus formed decomposed almost instantaneously. In order to insure complete removal of the xanthic acid, the acidified aliquot was extracted in a separatory funnel with two 20 c.c. portions of toluene (xanthic acid is insoluble in water and miscible with organic solvents). The aqueous layer of the extraction to which was added one water washing of the toluene layer was titrated after the addition of 5 c.c. of 0.2M K_2SO_4, with 0.002N iodine. A blank correction was applied as before. The applicability of this method to mixtures of potassium xanthate, sodium sulfite and sodium thiosulfate is illustrated below.

50 c.c. aliquots of a potassium xanthate mixed with either $Na_2S_2O_3$ or Na_2SO_3 solution were titrated with 0.002N iodine. Other 50 c.c. portions were acidified with 5 c.c. of 0.3M tartaric acid. These were then extracted with toluene as above. The aqueous layer was titrated with 0.002N iodine in each case.

The results of these experiments show that xanthic acid is completely removed by the toluene from the water layer so that in subsequent titration of the water layer only the inorganic ions such as sulfite, etc., when present, are titrated with iodine. Thus it is possible to differentiate between residual xanthate ion and other iodine-reducing ions.

Determination of Sulfate Ion.—A 100-c.c. aliquot of the filtrate was transferred to a steamed Pyrex Erlenmeyer flask acidified with 0.5 c.c. of 6M HCl and evaporated to about 15 c.c. on an electric hot plate, whereupon it was filtered and washed into a 50 c.c. steamed Pyrex beaker. The sulfate ion was then precipitated and weighed as $BaSO_4$ according to the micro method of Pregl.[8] Duplicate determinations agreed within about 2 per cent. Blanks on the potassium xanthate solution used were run along with the actual determination.

Determination of Alkalinity.—Because of the low buffering capacity of the filtrates, the pH was determined colorimetrically on 10 c.c. by means of the Klett-Beaver colorimeter.[9] Although precision within a few hundredths of a pH could be obtained on a single set of readings, duplicate determinations varied generally in the direction of a decrease in pH due probably to carbon dioxide from the breath of the operator and from the air. Accordingly, results are reported to the nearest tenth of a pH unit.

Qualitative Test of Sulfide Ion.—A 25-c.c. aliquot of the filtrate was tested for sulfide by the method involving a change of the sulfide ion to a blue thiazine dye.[10] This test was shown to be applicable in the presence of xanthate ion, at concentrations of as low as 0.06 mg. per liter of $S^{=}$ calculated as H_2S. This is equivalent to the sulfide ion that would result if the interaction of about 0.6 mg. per liter of potassium xanthate with lead sulfide took place.

From a series of runs the data given in Table 1 were obtained. The equations that were the basis for the calculation in equivalencies in columns 4 and 5 of Table 1 are:

$$PbS_mO_n + 2K^+ + 2X^- \rightarrow PbX_2 + (S_mO_n)^= + 2K^+$$
$$PbSO_4 + 2K^+ + 2X^- \rightarrow PbX_2 + SO_4^= + 2K^+$$
$$(S_mO_n)^= \equiv 2KX$$
$$(SO_4)^= \equiv 2KX$$
$$X \equiv \text{xanthate ion } (C = S.OC_2H_5S)^-$$

No sulfide ion was found in any of the above experiments. The failure to detect sulfide ion cannot be accounted for on the basis of its disappearance caused by the following interaction of sulfide with sulfite to form free sulfur, for this would go on only if supplied with sufficient hydrogen ion,[11] and experiments made with mixtures of sulfide and sulfite indicate that it does not go to any appreciable extent above pH of approximately 7.

Examination of the data shows that at the lower concentrations of potassium xanthate in the neighborhood of about 25 mg. per liter the removal of the xanthate ion can be accounted for by sulfate ion and reducing ions $(S_mO_n)^=$ thrown into solution. At the higher xanthate-ion concentrations the above ions do not account completely for the amount of xanthate ion abstracted and the mixture becomes progressively more

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[8] F. Pregl: Quantitative Organic Microanalysis, 139. Philadelphia, 1930. Blakiston and Son.

[9] J. J. Beaver: Optical Soc. Amer. (1929) **18,** 41.

[10] J. H. Yoe: Photometric Chemical Analysis, **1,** 373. New York, 1928. John Wiley & Sons.

[11] F. P. Treadwell and W. T. Hall· Analytical Chemistry, **1,** 372. New York, 1921. John Wiley & Sons.

TABLE 1.—*Metathetical Reactions between Galena and Xanthate Ion at Various Concentrations*

Experiment No.	Original Solution	Potassium Xanthate Removed, Mg. per 100 Grams Galena				Alkalinity	
	Potassium Xanthate, Mg. per Liter	Total	Accounted for as		Unaccounted for	Actual Filtrate, pH	Control Solution, pH
			Reduced Divalent Sulfur-oxygen Ions	Sulfate Ions			
A	16.3	12.5	2.5	10.9	0	7.0	6.3
B	23.2	17.7	2.8	12.6	2.3	7.2	6.4
C	32.4	23.6	3.1	12.8	7.7	7.7	6.5
D	65.0	30.2	3.0	12.7	14.5	8.5	6.6
E	129.2	31.4	3.3	12.9	15.2	8.8	6.8
F	162.6	32.8	3.6	13.1	16.1	9.0	
G	293.0	40.9	3.5	11.2	26.2	9.3	
H	592.0	39.5	3.5	11.1	24.9	9.4	8.1
I	1178	36.6				9.3	8.5
J	1645	38.8				9.3	
K	2620	38.8				9.2	8.6
New batch of galena.							
L	10.6	6.5	1.3	5.5	0	7.0	6.4
M	28.7	7.7	1.5	5.5	0.7	7.1	6.5
N	100	28.1	1.9	4.9	21.3	7.5	6.5
O	146	27.4	1.8	6.3	19.3	8.1	6.9
P	298	28.6	2.1	6.0	20.5	8.2	7.0

alkaline. The assumption may be made that the unaccounted-for xanthate ion is involved through metathetises, such as the following:

$$PbO + H_2O + 2[C{=\!=}S^{OC_2H_5S}_{S-}]^- \rightarrow Pb(C = S.OC_2H_5S)_2 + 2OH^- \qquad [1]$$

$$PbS + 2[C{=\!=}S^{OC_2H_5S}_{S-}]^- \rightarrow Pb(C = S.OC_2H_3S)_2 + S^= \qquad [2]$$

$$PbCO_3 + 2K^+ + 2[C{=\!=}S^{OC_2H_5S}_{S-}]^- \rightarrow Pb(C = S.OC_2H_5S)_2 + CO_3 + 2K^+$$

$$2K^+ + CO_3^= + H_2O \rightarrow OH^- + 2K^+ + H_2CO_3 \qquad [3]$$

Reaction 1 would be a possible explanation if the galena had associated with the oxide ion. This possibility can be rejected however, since the hydroxyl ion resulting from such a metathetical reaction would explain only a small part of the equivalent xanthate ion not accounted for by the other ions, even though there would be some slight buffering of the hydroxyl ion by the reducing ions.

Reaction 2 can be dismissed as a possibility on the basis of experiment. In no case was sulfide ion found in the filtrate from one of the above experiments, even though the sensitivity of the test was extremely great.

Reaction 3, where the hydroxyl from the ion hydrolysis of potassium carbonate, which in turn would come from the double decomposition of lead carbonate, associated with galena and xanthate ion, is possible. If an amount of potassium carbonate is taken such that its concentration is equivalent to that of the unaccounted for xanthate ion in Table 1, items G and H, its solution has a pH of 9.2 when measured colorimetrically.[12] This pH is about that developed with the high concentrations of xanthate on galena. The results are to be regarded as only approximate, however, as pH determinations in carbonate solutions of this concentration are difficult to obtain with any assurance of accuracy, though good precision can be obtained with care.

Metallic Ion of Xanthate Not Abstracted by Galena

During these interactions, the sodium or potassium ion concentration in the filtrate of whatever alkali xanthate is used with the galena does not change appreciably, showing that the alkali xanthate salts are not removed as such but only the xanthate ion is abstracted. This is proved by the following experimental work:

30 grams of galena (through 200 mesh) washed with water several times by decantation was shaken for 1 hr. with 300 c.c. of water containing 20 mg. of sodium xanthate. The galena was filtered off on an asbestos filter. 100 c.c. of the filtrate was acidified and evaporated carefully to dryness, then the residual sodium chloride was dissolved in 10 c.c. of water and the sodium determined by the method of Kolthoff and Barber.[13]

A similar experiment was carried out using 67 mg. of potassium xanthate in 300 c.c. of water. Potassium ion was determined in the filtrate by the micro method of Emich.[14] (The galena used was not of the same batch in both cases.)

The method used for the determination of sodium could be used over a range of from 0.6 to 5 mg. Na with an average precision of ±2 per cent. The method used for the determination of potassium could be used over a range from 1 to 10 mg. K with an average precision of ±1 per cent.

In the experiment with sodium xanthate the amount of that reagent removed as calculated from the iodimetric titration was 8 of the 20 mg. put into the system. The determined sodium ion in the original sodium xanthate solution corresponded to 20.4 mg. of sodium xanthate. After the treatment with galena the filtrate showed sodium ion equivalent to 20.6 mg. of sodium xanthate, which is no appreciable change. Obviously other ions took the place of the xanthate ions removed.

With potassium xanthate, by iodimetric determination 60.6 mg. of potassium xanthate went in, 35.2 mg. was abstracted. The potassium ion in the original amount of reagent was 61.2 mg., while that after the reaction corresponded to 60.6 mg. of potassium xanthate. The results are similar to those in previous experiments with the sodium xanthate.

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[12] Reference of footnote 9.

[13] H. H. Barber and I. M. Kolthoff: *Jnl.* Amer. Chem. Soc. (1928) **50,** 1625.

[14] F. Emich: Mikrochemisches Praktikum, 129. Munich, 1931. J. F. Bergmann.

ALKALINITY AND ITS CAUSE

The alkalinity that is developed in the interaction of higher concentrations of alkali xanthates and galena is also developed in the interaction of galena with other ions of widely different types which cannot themselves decompose to give hydroxyl ions. For example, a sample of galena that gave a filtrate after the galena xanthate interaction, with a pH of 9.2, gave a pH of 9.3 when a new sample was treated with potassium oxalate (104 mg. per liter) and a pH of 9.4 when still another sample was treated with potassium chromate (121 mg. per liter). Solutions of each of the original reagents had a pH of 6.8 to 7.1. Repetition of these experiments gave similar results. Oxalate and chromate like xanthate form insoluble lead salts.[15]

The indirect evidence that higher concentrations of alkali xanthate on galena gives rise to carbonate ion is corroborated further by the fact that powdered cerussite when treated with aqueous solution of potassium xanthate (300 c.c. of water and 60 mg. xanthate for 30 grams of cerussite) removes xanthate ion and the filtrate becomes alkaline (pH = 9.2).

That lead carbonate is actually associated with galena is demonstrated by the results of a direct microdetermination of the carbon dioxide liberated from the displaced carbonate ion.

A direct determination of carbonates in the small quantities that might be present in the filtrate from galena treated with aqueous potassium xanthate must necessarily be done under conditions where all possible chance of carbonate ion, or carbon dioxide contamination, can be avoided or accounted for. Briefly, galena ground in air to smaller than 200 mesh, as before, was introduced into a flask and shaken with potassium xanthate solution made up with CO_2 free water. The galena was filtered under conditions excluding carbon dioxide contamination. An aliquot portion of the filtrate was treated with acid to destroy xanthate ion. Because xanthates hydrolyze to carbonates[16] on boiling with water, especially in the presence of hydroxyl ion, means had to be taken to circumvent the possibility of contamination of the solution by carbonate ion from the source. Therefore the solution containing the filtrate from a treatment of galena with aqueous potassium xanthate was acidified with H_2SO_4 and allowed to stand 1 hr. in a closed system.[17] The liberated xanthic acid decomposed to carbon disulfide and alcohol according to the equation:[18]

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[15] It is interesting to note that the concentration of xanthate used in the flotation of sulfide ores is 0.1 lb. per ton, which with the water used is equivalent to 13 mg. per liter. In the technical use of xanthate for flotation, therefore, the concentration of the reagent is such that no alkalinity can be expected from the reaction of galena with xanthate ion. For other reasons, however, it is customary to work in slightly alkaline solution through the addition of lime.

[16] M. Ragg: *Chem. Zeit.* (1908) **32,** 630, 654, 677.

[17] For details see A. F. Knoll, Columbia Dissertation, 1932.

$$(C{=}S.OC_2H_5S)^- + H^+ \rightarrow \underset{H_2O \text{ insoluble}}{C{=}S.OC_2H_5SH} \rightarrow CS_2 + C_2H_5OH$$

In order to fix the CO_2 liberated from the carbonate ion in the filtrate by the acid, so that it could be concentrated, the solution in the closed system was made slightly alkaline with carbonate-free 0.5N alcoholic potash. A small amount of extraneous carbonate ion, which was allowed for by means of blank experiments, probably arose from the hydrolysis of carbon disulfide by hydroxyl ion:

$$CS_2 + 4OH^- \rightarrow CO_3^= + 2HS^- + H_2O$$

The alkaline carbonate solution was concentrated in the closed system, the solution cooled and acidified and the liberated CO_2 transferred to a Van Slyke gasometer,[19] to be measured volumetrically. Corrections were made for the extraneous CO_2 from the carbonate ion mentioned above, for the solubility of the CO_2 in the water and for other errors from blank determinations and from ones with known pure sodium carbonate in the system. From the amount of carbon dioxide obtained, the equivalent in terms of potassium xanthate was calculated:

$$PbCO_3 + 2K^+ + 2X^- = PbX_2 + 2K^+ + CO_3^=$$
$$(CO_3)^= \equiv CO_2 \equiv 2KX$$
$$X \equiv \text{xanthate radical}^{20}$$

Total reducing ions, that is, the sum of the unused excess xanthate ion and the new $(S_mO_n)^=$, were determined iodimetrically on an aliquot of the filtrate. After destruction of xanthate ion in another aliquot of the filtrate (by conversion into xanthic acid which decomposes into carbon bisulfide and alcohol),[21] the residual reducing ions $(S_mO_n)^=$ were determined by an iodimetric titration. Sulfate ion and pH were also determined on separate portions of the filtrate. The results are given in Table 2.

Apparently the carbonate associated with a given sample of galena is present initially with it and is constant in amount. When the concentration of the reacting xanthate reaches the point where the carbonate is displaced all of this ion is removed. No more seems to form as the galena stands exposed to air. On the other hand, the amount of other

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[18] H. Holban and A. Kirsch: *Ztsch. physik. Chem.* (1913) **82,** 325.

[19] D. D. Van Slyke: *Jnl. Biol. Chem.* (1917) **30,** 347.
J. F. McClendon: *Ibid.*, 259.

[20] Reference of footnote 17.

[21] Reference of footnote 18.

TABLE 2.—*Metathetical Reactions between Galena and Xanthate Ion in CO_2 Free System*

Experiment No.	Time of Exposure after Grinding	Potassium Xanthate Removed, Mg. per 100 Grams Galena				Alkalinity		
		Total	Accounted for as			Total $(S_mO_n)^- + (SO_4)^- + (CO_3)^-$	Actual Filtrate, pH	Original Solution, pH
			Reduced Divalent Ions $(S_mO_n)^-$	Sulfate Ion $(SO_4)^-$	Carbonate Ion $(CO_3)^-$			
A	initial	45.8	12.0	17.1	12.6	41.7	9.0	6.9
B	3 wks.	178	20	126	16	171	8.3	
New batch of galena.								
C	initial	39.0	8.0	5.8	26	39.9	9.3	6.8
D	1 wk.	52.3	12.7	12.8	(26)[a]	51	9.2	
E	2 wks.	58.3	13.8	16.1	27.2	57.1	9.3	
F	3 wks.	64.8	14.1	18.7	25.8	57.6	9.5	
New batch of galena exposed to air about one month.								
G		87.0	19.2	26.4	[a]		9.8+	
H		88.8	19.6	27.1	41	87	9.8+	

[a] Determinations spoiled. Orig. conc. potass. xanthate 200 mg. per liter.

ions displaced by xanthate increases on standing. This phenomenon will be discussed in a later section.

SULFIDE IONS NOT INVOLVED

The amount of xanthate ion abstracted by galena can be accounted for now on the basis of metathetical reactions between the potassium xanthate and lead sulfate, other lead-sulfur-oxygen compounds and lead carbonate associated with the galena. There is *no indication* that reaction takes place between xanthate ion and lead sulfide itself. Apparently the sulfate ion is displaced from the surface first, then the somewhat less soluble sulfur-oxygen reducing ions, and finally the carbonate ion. This is in agreement with order of the solubility products of lead salts of these ions, if we assume that this product holds at least qualitatively for these salts when associated with the galena crystal lattice. Because of the small solubility product of lead sulfide, the amount of sulfide ion, if any, that comes from the interaction of the potassium xanthate and lead sulfide is below the detectable limit.

EFFECTS OF LARGE EXCESS OF ALKALI XANTHATE

The falling off in the amount of xanthate ion abstracted by galena from the solutions of higher concentrations of potassium xanthate is evidently due to the formation of soluble complex ions. As a matter of fact, the otherwise insoluble crystals of pure precipitated lead xanthate disappear readily when shaken with an excess of a concentrated aqueous solution of potassium xanthate. Apparently this is the same phenomenon as that referred to above. In connection with this it may be noted that the "captive bubble test"[22] shows smaller and smaller bubble attachment at the treated galena surface as the xanthate concentration of the solution used to treat the galena is increased, until finally it is negligible.

Two Types of Lead-oxygen Ions Associated with Galena

If powdered galena which has been exposed to filtered air for several hours is treated for long periods with water, lead sulfate, sulfite and other lead-sulfur-oxygen salts are dissolved off until the solution apparently is saturated with these salts. Both the negative radicals on one hand, and the lead ions on the other, may be formed and the former are approximately equivalent to the latter. In the case of lead sulfate, the amount formed (42.8 mg. per liter at 23° C.) corresponds to the solubility of ordinary orthorhombic lead sulfate (41 mg. per liter at 20° C., 45 mg. per liter at 25° C.).[23]

The reducing ions obtained in shaking (1) 20 grams of oxidized galena with 200 c.c. of water, (2) 50 grams of galena with 200 c.c. of water were equivalent (after 2 hr. shaking) to:

(1) 29.4 c.c. of 0.002N I per liter,
(2) 30.0 c.c. of 0.002N I per liter.

It is, of course, impossible to designate these ions as sulfite, thiosulfite, etc., but the mixture (no sulfide) is constant and a determination of the lead ion shows that there is enough of these metal ions over and above those necessary to pair off with the sulfate, to be allocated to these sulfur-oxygen reducing ions.

Newly ground galena, however, gives smaller values for the solubility of the lead sulfate and the other lead-sulfur oxygen compounds associated with it, and in this case sulfate, sulfite and other lead-oxygen ions may be displaced from galena by xanthate in amounts in excess of that which can be dissolved off with water alone. The following experiment illustrates this.

20-gram portions of galena (through 200 mesh) were shaken with 200 c.c. of water for increasing time periods. Reducing ions measured in terms of $\frac{N}{500}$ iodine and sulfate ion (calculated as $PbSO_4$) were determined on aliquot portions of the filtrate. At the same time, 30-gram portions of the galena were shaken with aqueous potassium xanthate (200 mg. per liter) in order to determine (by displacement of the anions) approximately the total quantity of soluble lead salt associated with the galena. This was done by determining the sulfate and sulfur-oxygen-reducing ions in the filtrate from the galena-xanthate mixture. The results are summarized in Table 3.

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[22] Reference of footnote 1.

[23] A. Seidell: Solubilities of Inorganic and Organic Substances. New York, 1919. D. Van Nostrand.

TABLE 3.—*Lead Salts Dissolved by Water and Displaced by Xanthate Ion from Galena*

Time, Hr.	Reducing Ions, $\frac{N}{500}$ Iodine per Liter, c.c.		$PbSO_4$, Mg. per Liter	
	Dissolved by Water	Displaced by Xanthate Ion	Dissolved by Water	Displaced by Xanthate Ion
½	3.0	24.2	9.9	12.5
1	4.0		9.5	
2	4.2		9.6	
4	4.1	28.2	9.8	13.2
Another batch of galena.				
			9	28
1	3.0	13	10	29
1	3.0	15	6.5	10
1	2.0	14	11	20
1	4.2	13	6.0	8.0

While the experimental data given can by no means be regarded as tonclusive, the indications are, from the above experiments, that the compounds formed by the initial oxidation of galena are more firmly anchored to the surface of the galena particle, and differ in structure from the compounds in their normal state. It is interesting to note in this connection that Menzies and Sloat[24] have found that the evaporation of solutions of salts on a clean galena surface results in orientation of the salt being deposited if the latter is isomorphous with the galena (sodium chloride, for example) whereas if the salt is not isomorphous with the galena no orientation takes place. In the light of the experimental work reported here, and of the experiments of Taggart, Taylor and Ince,[25] it is highly probable that the surfaces used by Menzies had undergone some oxidation. If this is so, the conclusion follows that the lead-sulfur-oxygen compounds (lead sulfate, etc.) first formed are probably isomorphous with the galena. It might be expected also that this anchored "cubic" lead sulfate would have a different solubility from the common (orthorhombic) lead sulfate. The solubility might be expected to be intermediate between lead sulfide and lead sulfate; in other words, less than the solubility of common orthorhombic lead sulfate.

On continued oxidation, the lead-sulfur-oxygen compounds (being farther removed from the surface of the lead sulfide and therefore from its sphere of influence) change over into their normal orthorhombic form.

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[24] C. A. Sloat and W. C. A. Menzies: *Jnl. Phys. Chem.* (1931) **35**, 2008.

[25] Reference of footnote 1.

This is indicated by the fact that highly oxidized galena surfaces give normal solubility for lead sulfate.

It is probable that these adherent "cubic" lead-sulfur-oxygen compounds are the ones that are most effective in attaching and orienting the xanthate ion to the crystal lattice of galena.

The fact that the amounts of xanthate ion removed by freshly ground galena (through 200 mesh) on several different batches were fairly constant in amount seems to preclude the possibility of irregular contamination by inclusions. Different samples of freshly ground galena, for example, abstracted 40, 37, 45, 39 and 34 mg. of xanthate ion (calculated as potassium xanthate) per 100 grams of galena.

Source of Ions Associated With Galena

In the previous experiments on carbonate ion it was implied that the amount of this ion associated with a batch of galena is constant but that the amount of other ions formed on the galena surface was a function of time of exposure to air. The rate of oxidation of a sample of powdered galena in air can be followed by noting the abstraction of xanthate.[26]

For example, the abstraction of xanthate initially on a 100-mg. sample of powdered galena was 24.1 mg., which became successively 25.3 for 3 hr.; 35.9 for 1 day; 41.1 for 2 days; 41 for 3 days; 48.0 for 7 days; 53.2 for 16 days; and 60.5 for 32 days exposure to CO_2 free air. The pH of the filtrate remained at about 9.3 throughout.

The amounts of sulfate and iodine-reducing sulfur-oxygen ions increased steadily during this time but the carbonate ion and concurrent alkalinity remained substantially constant. Even when the powdered galena is exposed to ordinary air laden with the usual amount of carbon dioxide, there is no appreciable increase in the amount of carbonate ion displaced by the xanthate.

If the massive galena is dry ground in nitrogen instead of in air, there is a great decrease, as might be expected, in the subsequent xanthate removed by the sample when compared with an air-ground sample. After one hour's grinding in air the abstraction was about 16 mg. per 100 grams of galena compared to 5.5 mg. of xanthate with nitrogen-ground galena. Wet grinding under corresponding conditions also shows that oxidation is rapid in air.

Orientation of Sulphydryl Radical

Turning to the xanthate radical that takes part in these reactions, it follows that that portion of the xanthate ion that displaces the "anchored" sulfate and sulfite becomes itself firmly anchored and thereby oriented.

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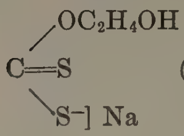
[26] Since iodine-consuming ions such as sulfite, etc., are thrown into solution as the xanthate is abstracted, a direct iodimetric titration will often show no apparent xanthate removal. This must be considered and allowed for as in previous instances.

It is this film that is water-repellent, the remainder of the xanthate that is removed having nothing to do with the alteration of the surface of the galena. So firmly is the xanthate radical anchored that washing in running water does not remove it.

If we assume that the water-repellent capacity of the lead xanthate lies in the ethyl grouping, a change in the character of this grouping, from a water-repellent type to one that is water avid should result in change in the character of the surface. The hydroxyl (—OH) grouping is one that carries with it water-avid properties, as evidenced by the fact that organic compounds assigned this grouping are characterized (other things being equal), for example, by greater solubility in water than one not assigned this grouping.

Accordingly, an attempt was made to prepare a compound having a hydroxyl group in place of one of one hydrogen atom in the ethyl grouping:



$$C(OC_2H_4OH)(=S)S^-]\ Na$$

(a glycol xanthate)

5 grams of sodium was dissolved in an amount of redistilled ethylene glycol in excess of that necessary to give the monosodium derivative of ethylene glycol.[27] The rather viscous, clear, pale yellow liquid resulting was shaken with an excess of carbon bisulfide (25 c.c.) for 10 hours:

$$C_2H_4(OH)_2 + Na = C_2H_4 \cdot OH \cdot ONa$$
$$C_2H_4 \cdot OH \cdot ONa + CS_2 = [(CS)(OC_2H_4OH)S]\ Na$$

Some of the carbon disulfide evidently reacted with the monosodium glycoxide, as the glycol layer slowly changed to a deep orange color. The mixture of carbon disulfide and glycol was extracted with ethyl ether to remove the excess carbon disulfide. The mixture of glycol and what was presumably the sodium salt of glycol xanthic acid was soluble in water. The fact that the aqueous solution was only slightly alkaline indicated that practically complete reaction took place between the carbon disulfide and the monosodium glycoxide. The sodium salt of glycol xanthic acid could not be separated from the glycol mixture.

Solutions of the presumed glycol xanthate gave a red precipitate with lead ion and a white precipitate with zinc ion. A few drops of the "glycol xanthate" were dissolved in 1 liter of water and 300 c.c. of this were shaken with 30 grams of galena. No collection of a froth containing galena particle took place at the surface of the solution. This indicated that the surface of the galena had changed little or not at all in wetability. (The small amount of glycol present would have no effect). That some of the "glycol xanthate" had been abstracted by the galena is evidenced by the following iodine titration.

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[27] A. Wurtz: *Ann. Chem.* [3] (1859) **55**, 429.

"Glycol xanthate" solution before shaking with galena used 170 c.c. 0.002N iodine per liter of solution; after shaking with galena = 117 c.c. 0.002N iodine.

An ethyl xanthate solution of the equivalent concentration would have been affected to about the same extent.

As further evidence of the effect of the hydrocarbon grouping of the xanthyl radical on the degree to which this ion changes the wetability of minerals, Wark and Cox[28] report that contact angle between the mineral and water increases (the mineral becomes less wetable) as the length of the hydrocarbon chain increases; for example, a butyl xanthate gives a higher contact angle than an ethyl xanthate when used in equivalent concentrations.

Stability of Alkali Xanthates

Because of the confusion of data on the stability of the alkali xanxhates, and because in certain types of decomposition of the alkali oanthates alkalinity is developed, and further because the possible develepment of alkalinity from the xanthate has bearing on the results of the txperiments, it was thought desirable to include a short discussion of the reagent here.

Potassium ethyl xanthate may be taken as the example. It was prepared in the usual way,[29] recrystallized twice from alcohol and kept in a vacuum desiccator over concentrated sulfuric acid for 24 hr. The salt is almost white and nearly odorless. Its water solution has a pH = 7.2 and its conductivity shows that xanthic acid is a strong acid. The precipitated lead salt dried at 60° C. contained 45.8 ± 0.005 per cent lead against 46.1 per cent theoretical. Iodine titration, 99.5 per cent ± 0.10 per cent xanthate.

As pointed out by Von Holban and Kirsch[30] and others, alkali xanthates do decompose when acidified to give CS_2 and alcohol, but it has been found that the pH must be less than 5 and the temperature above 5° C. for this to happen. Below this temperature, the very water-insoluble xanthic acid is stable (and may be extracted with organic solvents). To carbon bisulfide and alcohol produced in this way, and by reaction 8 below, Kellerman and Bender[31] ascribe the surface alteration of galena. That CS_2 is not present in sufficient quantity to alter the surface properties of galena comes also from the work of Wark and Cox,[32] who find that carbon bisulfide has no measurable effect on the contact angle between galena and water, until the amount of carbon bisulfide added to the water exceeds its solubility, whereupon a visible coating of carbon bisulfide on the galena results. It is well known that liquids like carbon disulfide, which have a high interfacial tension measured against water, and oily substances in general, when applied to solids, raise materially the contact angle between the solid and water.[33] In an alkaline solution (1 to 2N) according to Ragg,[34] there is extensive decomposition of xanthate ion when judged by the composition of the insoluble cuprous salts which precipitate from the decomposed solutions and which are probably in the main thiocarbonates.[35] This alkalinity is beyond that reached in any of the present experiments and again beyond that in usual flotation practice.

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[28] I. W. Wark and A. B. Cox: Page 189, this volume.

[29] P.-J. Beilstein: Organische Chemie (1921) **3**, 209.

[30] Reference of footnote 18.

[31] K. Kellerman and E. Bender: *Koll. Ztsch.* (1930) **52**, 240.

[32] Reference of footnote 28.

The equations describing the most likely reactions by which alkali xanthates may decompose are:[36]

$$(C{=}S.OC_2H_5S)^- + H^+ \rightarrow CS_2 + C_2H_5OH \qquad [4]$$

$$(C{=}S.OC_2H_5S)Na + NaOH \rightarrow C{=}S.ONa.SNa + C_2H_5OH \qquad [5]$$

$$(C{=}S.OC_2H_5S)Na + 2NaOH \rightarrow C{=}S.(ONa)_2 + NaSH + C_2H_5OH \qquad [6]$$

$$(C{=}S.OC_2H_5S)Na + NaSH \rightarrow C{=}S.(SNa)_2 + C_2H_5OH \qquad [7]$$

$$(C{=}S.OC_2H_5S)Na + HOH \rightarrow CS_2 + C_2H_5OH + Na^+ + OH^- \qquad [8]$$

$$4(C{=}S.OC_2H_5S)Na + 2H_2O + O_2 \rightarrow 2\underset{\text{dixanthogen}}{(C_2H_5O.C.S.S.S.CSOC_2H_5)} + 4OH^- \qquad [9]$$

According to experience in this laboratory, the only commonly met decompositions are those taking place around the neutral point; namely those recorded in equations (8) and (9). The determinations to prove this follow:

1. Iodimetric titration will detect changes as in equations 4 and 8, since nothing except xanthate is affected by iodine.

2. Iodimetric determination of the aqueous layer after acidification and extraction with toluene will detect sulfide ion. Unchanged xanthate on acidification is either destroyed to give non-iodine-consuming substances (equation 4) or xanthic acid, which is very soluble in toluene. Sulfide ion if present would be titrated in the water layer.

3. Determination of pH colorimetrically[37] would indicate any reaction giving hydroxyl ion either directly as in equations 4 and 9, or indirectly.

4. Qualitative test for sulfide ion[38] would show reaction.

5. Color of precipitate on adding lead ion is white for lead xanthate; for thiocarbonates brown, and for sulfide, black. The amount of lead xanthate precipitated also serves as a guide.

The results are given in Table 4.

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[33] A. F. Taggart: Handbook of Ore Dressing, 777. New York, 1927. John Wiley & Sons.

[34, 35, 36] Reference of footnote 16.

[37] Reference of footnote 9.

[38] Reference of footnote 10.

TABLE 4.—*Stability of Potassium Xanthate Solution at pH 7±*
1.1526 grams per liter

	Initial ± ¼ hr.	1 hr.	2.5 hr.	3 hr.	5 hr.	7 hr.
Iodine titer on 10 c.c. direct	38.28	38.28	38.33	38.23	38.26	38.03
Iodine titer on 10 c.c. after acidification and toluene extraction	0.03	0.02	0.05	0.05	0.08	0.10
Weight of lead xanthate 50 c.c. (Calc. = 0.08023) based on analysis	0.07996	0.08003 3 days	0.07996 0.07629	0.07971 (buff colored)		0.07921
Colorimetric pH	6.5	7.5	8.5	8.5	8.6	8.4
Color of lead precipitate	white	white	white	white	white	white
Qual. test $S^=$	0	0	0	0	0	0

At pH = 8, and at 10, the results are substantially the same, showing that the decomposition of alkali xanthate in water solution is slow. The principal reaction is probably the one of oxidation to dixanthogen.

SUMMARY

1. The action of alkali xanthates with galena is one of ionic interchange. The alkalinity developed in the interaction is due to lead carbonate.
2. Alkali xanthate is not adsorbed as such.
3. Sulfide ion from the galena crystals does not take part in the interaction.
4. A set of sulfur-oxygen ions closely related to the galena crystal is postulated as being present. These are displaced by the sulphydryl radical. These "anchored" lead compounds have a solubility in water different from the gross form of the same salts.
5. By virtue of the displacement of these anchored sulfur-oxygen ions, the xanthate ion is oriented.
6. The stability of alkali xanthates is discussed.

A Study of Slime-coatings in Flotation

By Guido R. M. del Giudice,* Member A.I.M.E.

(New York Meeting, February, 1934)

The term "slime-coating" is not new in the art of flotation; the phenomenon has been observed and described by Taggart;(1)† Taggart, Taylor, and Ince;(2) and by Ince.(3) Notwithstanding that flotation was introduced for the treatment of finely crushed ore, the adverse effects of slimes have been recognized since the earliest days of the process, and the Elmore, Delprat, Potter, and other processes actually specified desliming of the ore prior to treatment. The cause of this trouble has been studied and described by Taggart;(1) Taggart, Taylor, and Ince;(2) Ince;(3) Morrow and Griswold;(4) and by Gaudin and Malozemoff.(5)

Old Theory of Slime-coatings

At present, so far as published work shows, there seems to be but one theory for the mechanism of slime-coatings. Ince(3) states that they are due to attraction between electrostatic charges of opposite sign on the sulfide and on the slime particles, supporting this theory by migration experiments in the Burton tube. He also used the microscope in conjunction with a glass cell employed by Beans and Eastlack(6) in studying migration, but met with interference from endosmosis.

Several attempts have been made in the Department of Mineral Dressing at Columbia University to duplicate Ince's experiments with the Burton tube, but the results have never been convincing. Formation of precipitates which settled through the pulp levels, and evolution of gas at the electrodes, which disturbed the interface between the pulp and the distilled water above it, seriously interfered with observations. However, by use of the migration cell shown in Fig. 1, endosmosis effects and the disturbances due to precipitation and gas evolution were avoided, and distinct and reproducible migrations or absence of migration were observed.

The cell was made of bakelite and had platinum electrodes. Objectives were employed which had a working distance of at least three millimeters,[1] and the parts of the mounting which came in contact with the suspension were covered with a film of purified paraffin. A voltage of 220 was impressed at the electrodes.

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† Reference numbers in parentheses refer to bibliography at end of paper.

[1] The use of an objective having a relatively large working distance excludes from the field of observation those particles closer to the lens than the working distance,

Using this apparatus, it was found that quartz in distilled water and in 0.001-molar sodium carbonate solution migrated towards the positive electrode; and that iron oxychloride and iron oxybromide sols migrated towards the negative electrode. However, with galena in distilled water and in 0.001-molar carbonate solution, with sphalerite in distilled water, and with calcite in distilled water, the results were doubtful.

Ince⁽³⁾ found that, in distilled water, galena, sphalerite, and quartz all migrated towards the positive electrode; yet, in distilled water, sphalerite slime and quartz slime coat galena readily. In view of these apparent inconsistencies, and also of the fact that Ince found flocculation of the slimes to concur with coating, and further of the failure to obtain definite results with the migration cell, it was decided to abandon the electrostatic charge theory completely and to attack the problem from the standpoint of chemical reaction.

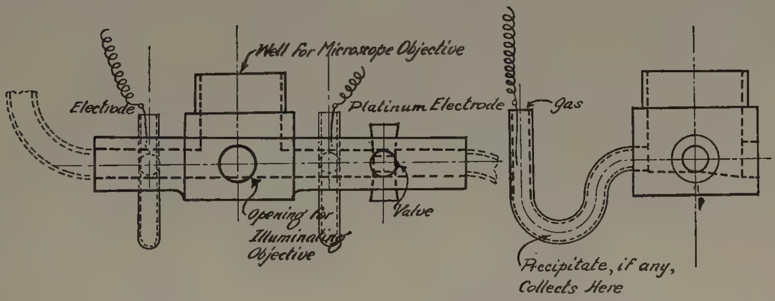
Well for Microscope Objective
Electrode
Platinum Electrode
Gas
Opening for Illuminating Objective
Valve
Precipitate, if any, Collects Here

Fig. 1.—Apparatus for observing migration.

Chemical Hypothesis of Slime-coatings

Taggart, Taylor, and Knoll,⁽⁷⁾ also Taylor and Knoll⁽⁸⁾ have shown that sulfide mineral surfaces become oxidized; and Taggart, Taylor, and Knoll,⁽⁷⁾ have postulated that these oxidation products ionize in water and, if within a certain low solubility range (approximately 1 to 35 mg. per liter), give a free-swimming ion and another which remains anchored in the lattice of the mineral. Any "charge" present on the particle, therefore, is probably due to the anchored ion.

In the study of soluble collecting reagents, these experimenters obtained data which indicated that there is a simple chemical reaction—metathesis, in the cases investigated—between the metal ion from the oxidized sulfide surface and the organic acid radical of the collective reagent. Reasoning by analogy, Professor Taggart has postulated in class lectures for several years that slime particles of gangue are cemented to

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and thereby avoids obscuring a true migration by any endosmotic effect of the objectives themselves.

sulfide mineral surfaces through a reaction between the anchored ions of the two minerals.

1. A clean, cleavage surface and a clean, polished surface of galena were placed in a pulp composed of 50 grams of −200-mesh calcite and 200 c.c. of boiled, distilled water at normal temperature. The pulp was agitated for 2 min. The galena particles were then taken out of the pulp, washed in three separate baths of distilled water, dried in a stream of air from an electric fan, and examined under the microscope. The two surfaces were similarly coated with slime.[2] Figs. 2 and 3 are photomicrographs of the polished galena surface, under different kinds of illumination.

That the specks appearing in the photographs are not depressions in the galena surface is proved by the photograph taken with the Ultropaque.[3] These particles cannot be lead carbonate precipitated upon the galena from solution, for a galena surface treated for 5 min. in sodium carbonate solution should show a surface alteration under the microscope; but this is not the case. Eliminating depressions in the mineral surface and precipitated lead carbonate, the only remaining possibility is that the specks are calcite particles. This conclusion is confirmed by examination of the surface with a petrological microscope, using crossed nicols with vertical illumination and a ¼λ gypsum plate. Upon rotating the stage holding the specimen, the isotropic galena surface retains the characteristic pink color of the compensator, while the specks exhibit the familiar color changes of anisotropic calcite. Finally, if a drop of dilute hydrochloric acid be placed upon the slime-covered surface, and the surface be examined with a low-power microscope, small bubbles of gas may be seen to evolve from the specks.

Applying the chemical hypothesis, the calcite slime particles were cemented to the galena surface by lead carbonate precipitated according to the following reaction:[4]

$$\underset{\substack{\text{surface}\\\text{of}\\\text{galena}}}{PbSO_4} + \underset{\text{slime}}{CaCO_3} \rightarrow \underset{\text{cement}}{PbCO_3} + \underset{\text{in solution}}{CaSO_4}$$

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[2] The purpose of testing both kinds of surface was to establish similarity so that polished surfaces, which are more easily cleaned and photographed, could be used in subsequent tests.

[3] In taking the photomicrograph with the Ultropaque, the light from the annular condenser was focussed on a plane slightly above the surface of the galena. The fact that the specks appear bright while the galena remains dark therefore indicates that the specks are projections above the surface of the mineral. If the specks were depressions, they could not appear bright unless the light from the annular condenser were focussed on a plane below the surface of the mineral.

[4] In this paper, lead sulfate is used as a generic term to denote the lead-oxygen-sulfur compounds that are present on the surface of galena.(7,8)

or as shown diagrammatically in Fig. 4, without implication as to shapes and relative sizes.

This reaction is indicated by a consideration of the published solubilities of the compounds involved.(9,10) Thus, lead carbonate, having a solubility of 1.7 mg. per liter of water, will be formed because it is less soluble than lead sulfate (42 mg. per liter of water) or calcium carbonate (65 mg. per liter of water).[5]

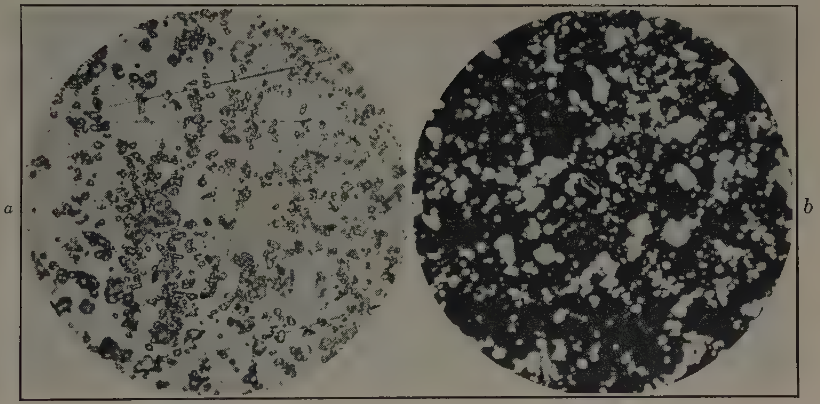

FIG. 2. FIG. 3.

FIGS. 2–3.—GALENA COATED WITH CALCITE SLIME.
a. Vertical illumination. × 500. *b.* Ultropaque illumination. × 450.

2. A 50-gram portion of −200-mesh galena was placed in 300 c.c. of boiled, distilled water at normal temperature, shaken for 15 min., and filtered. The filtrate was found to contain 7.5 mg. of sulfate ion per liter of water.(11)[6]

Taylor and Knoll(8) have shown that these sulfate ions come from the galena surface oxidized during and after grinding.

3. A 50-gram portion of −200-mesh calcite was shaken with 300 c.c. of boiled, distilled water at normal temperature, and filtered. The filtrate, which was found to contain carbonate ion,[7] was then added to a 50-gram portion of −200-mesh galena, shaken for 15 min., and filtered. The filtrate was found to contain 9.9 mg. of sulfate ion per liter of water.

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[5] Tabular values of solubility are always to be scanned with suspicion and used with discretion. However, with respect to the figures herein quoted a number of values from different sources are of the same relative magnitude.

[6] This amount of sulfate ion is equivalent to 23.8 mg. of lead sulfate per liter, which is well below the available figure for the solubility of lead sulfate in water (42 mg. per liter of water). This is in accord with Taylor and Knoll(8) who have postulated that compounds formed at the surface of galena may be expected to have a solubility intermediate between that of lead sulfide and that of the surface compound in bulk.

[7] A white precipitate which was obtained with lead acetate showed, under the microscope, the characteristic pseudohexagonal, branched rods of lead carbonate.

4. A mixture of 50 grams of −200-mesh galena and 50 grams of −200-mesh calcite was similarly shaken with 300 c.c. of boiled, distilled water at normal temperature, and filtered. The filtrate was found to contain 15.9 mg. of sulfate ion per liter of water.

The above results may be correlated thus:

A.—Filtrate from galena shaken with water. (Exp. 2.)......	7.5 mg. of sulfate ion per liter of water.
B.—Filtrate from galena shaken in filtrate from calcite shaken with water. (Exp. 3.)	9.9 mg. of sulfate ion per liter of water.
C.—Filtrate from mixture of galena and calcite shaken with water. (Exp. 4.)	15.9 mg. of sulfate ion per liter of water.

The increase of sulfate ions in B as compared with A indicates a metathetical reaction between lead sulfate from the oxidized galena surface and the carbonate ion present in the filtrate from the calcite shaken with water. The increase of sulfate ion in C as compared with B indicates a reaction between the oxidized galena surface and a greater concentration of carbonate ions. The latter may be due either to ionization of calcium carbonate that feeds into the solution with removal of carbonate ion, to maintain equilibrium, thus:

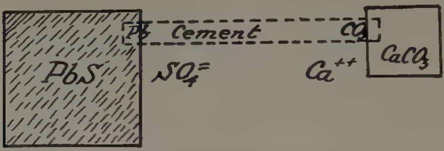

Fig. 4.—Diagram of chemical reaction between oxidized galena and calcite.

$$CaCO_3\ (\text{solid}) \rightleftharpoons CaCO_3\ (\text{sol.}) \rightleftharpoons Ca^{++} + CO_3^{--}$$

or to carbonate ion anchored at the calcite surface (see Fig. 4), or to both.

5. A slime pulp was prepared as follows: A 50-gram portion of −200-mesh calcite was added to 200 c.c. of 0.1 normal lead acetate solution, shaken for 15 min., filtered, and thoroughly washed. The calcite thus prepared was then placed in 200 c.c. of boiled, distilled water at normal temperature, and in this pulp a clean, polished surface of galena was treated for 15 min. The galena particle was then removed from the pulp, washed in three baths of distilled water, and dried. Microscopic examination of the galena surface showed very little slime-coating.

6. A clean piece of calcite was treated for 15 min. in 0.1 normal lead acetate solution. It was then washed thoroughly in distilled water and finally shaken for 15 min. in water saturated with hydrogen sulfide. The calcite particle was subsequently removed, washed again in distilled water and dried. Microscopic examination of this calcite particle showed that different parts of the surface had become black.

Apparently the following reactions had occurred:

$$Pb(C_2H_3O_2)_2 + CaCO_3 \rightarrow PbCO_3 + Ca(C_2H_3O_2)_2$$
$$PbCO_3 + S^{--} \rightarrow \underset{\text{Black}}{PbS} + CO_3^{--}$$

Lead sulfide, with a solubility of 0.3 mg. per liter of water,(9,10) was probably formed.

From experiments 5 and 6, it appears that calcite slime particles can be coated with lead carbonate, thus closing the calcite surface and thereby preventing calcite from coating a galena surface.

7. A clean, polished surface of galena was placed in a pulp composed of 50 grams of −200-mesh fluorite and 200 c.c. of boiled, distilled water at normal temperature. The pulp was agitated for 5 min. The galena particle was then taken out of the pulp, washed in three separate baths of distilled water, dried in a stream of air from an electric fan, and examined under the microscope. The galena surface was but slightly coated with slime.

Fluorite (CaF_2), having a solubility of 16 mg. per liter of water,(9,10) will not coat galena because it is less soluble than lead fluoride (640 mg. per liter of water) and also less soluble than calcium sulfate (1790 mg. per liter of water).

8. A pulp was prepared by adding 50 grams of −200-mesh fluorite to 200 c.c. of a solution containing 1 gram per liter of sodium oxalate. The mixture was shaken for 15 min., filtered, and then the fluorite slime was thoroughly washed. The fluorite thus prepared was then placed in 200 c.c. of boiled, distilled water at normal temperature, and in this pulp a clean, polished surface of galena was treated for 5 min. The galena particle was then removed, washed and dried, and finally examined under the microscope. The galena surface was coated with slime.

Apparently the following reactions had occurred:

$$\underset{\text{Fluorite}}{CaF_2} + Na_2C_2O_4 \rightarrow 2NaF + \underset{\text{At surface of fluorite}}{CaC_2O_4}$$

Calcium oxalate, having a solubility of 9 mg. per liter of water,(9,10) will be formed at the surface of fluorite because it is less soluble than calcium fluoride. This treated fluorite will coat galena because the solubility of the lead oxalate cement (1.5 mg. per liter of water) is less than that of either calcium oxalate or calcium sulfate.

The results of experiments 1 to 8 are all in accord with the chemical hypothesis of slime-coatings. Additional experiments, described under "Prevention of Slime-coating" give further evidence in support of that hypothesis.

9. Using the technique developed by Wark and Cox,(12) polished surfaces of galena which had been coated by calcite slime were tested with a bubble machine(2) in a solution containing 25 mg. of potassium

ethyl xanthate per liter of water, at a pH below the critical value.(13) No contact angle developed after 15 minutes.[8]

Apparently, the calcite slime-coating makes a galena surface behave substantially like a calcite surface. The following experiment was conducted to check the performance of the bubble machine.

10. An artificial mixture composed of 20 grams of −65 + 100-mesh galena and 30 grams of −65-mesh calcite was placed in a 50-gram Utah experimental flotation machine(14) with 0.04 lb. of potassium ethyl xanthate and 0.075 lb. of steam-distilled pine oil per ton, both reckoned on the solid. The froth was collected until no more galena came up with it. The concentrate and the tailing were immediately passed through an elutriator several times to remove suspended slime. Microscopic examination of the galena particles from the concentrate showed no slime-coating (Fig. 5), but the galena particles which remained in the tailing did show slime-coatings on their surfaces (Fig. 6).[9]

Further experimentation showed that, in distilled water, a clean galena surface may be coated by quartz, sphalerite, rhodochrosite, and galena slimes, as well as by calcite (Fig. 7), which coatings may be explained by the chemical hypothesis as follows:

Sphalerite (ZnS), having a solubility of 7 mg. per liter of water, reacts with lead sulfate (solubility, 42 mg. per liter of water) on the surface of galena, producing lead sulfide (solubility, 0.3 mg. per liter of water) which acts as the required cement.

Rhodochrosite ($MnCO_3$), the solubility of which is 65 mg. per liter of water, reacts like calcite in producing lead carbonate which then serves as cement.

Quartz (SiO_2) in water standing in ordinary laboratory glass probably has a surface coating of ionized sodium silicate,(7) and this reacts with lead sulfate to produce lead silicate as the cement.

The reason for the coating of galena by galena is not altogether clear. However, the fact that water glass (sodium silicate) prevents the coating of galena by galena seems to indicate that the coating may be due to the

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[8] Galena readily gives a contact angle of 60° in a solution containing 25 mg. of potassium ethyl xanthate per liter of water, whereas calcite gives no contact.(12)

[9] To the author's knowledge, no data are available to account for this difference in behavior between different particles of the same mineral (galena) in the pulp. Wark and Cox, faced with a somewhat similar difficulty, postulated a scouring effect in the flotation machine as the means of cleaning surfaces that would otherwise be slime-coated. This explanation, although difficult to substantiate, might possibly apply here. A more reasonable explanation, it would seem to the author, lies in the distinct possibility of a difference in the extent of oxidation on the galena surfaces in the pulp. The galena was broken down from a large lump, the outer surfaces and the cleavage cracks of which would probably be more highly oxidized than the fresh surfaces produced in the grinding.

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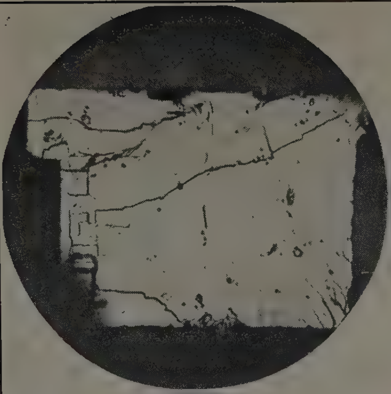

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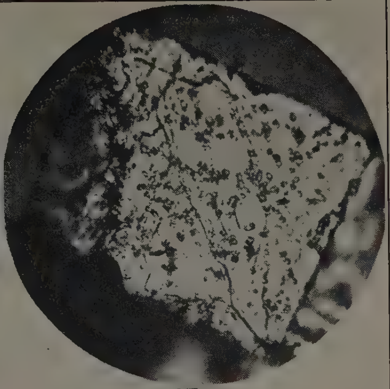

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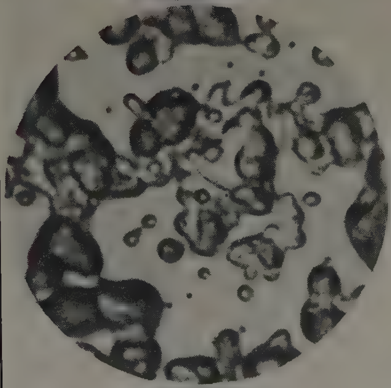

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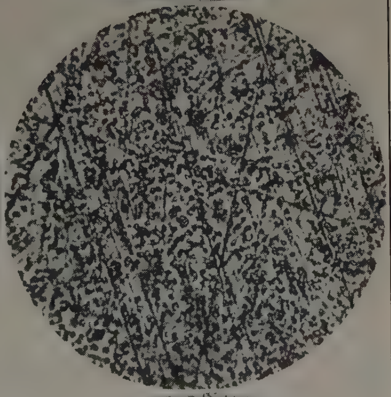

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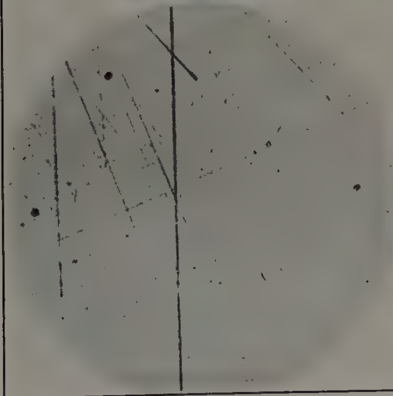

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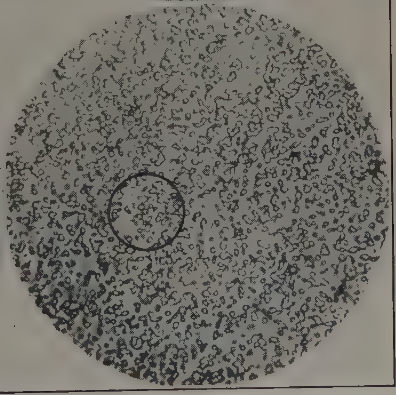

Fig. 5.—Uncoated galena particle from the flotation concentrate. × 500.

Fig. 6.—Coated galena particle from the flotation tailing. × 500.

Fig. 7.—Quartz slime coating on galena surface. × 1700.

Fig. 8.—Galena surface treated for 15 min. in calcite slime. × 500.

Fig. 9.—Galena surface treated for 15 min. in calcite slime and water glass (2 lb. per ton of solid). × 500.

Fig. 10.—Calcite coating on galena.

presence of some unknown impurity which enters into the formation of the cement along the lines predicated in this paper.

PREVENTION OF SLIME-COATING

The chemical hypothesis of slime-coatings likewise suggests means for preventing them. Indeed, the ability to predict that certain reagents will inhibit slime-coatings is additional evidence in support of the chemical hypothesis. Briefly, an inhibitor of slime-coatings should be a reagent which can diminish the solubility at the surface of the mineral that we are attempting to float to less than that of the cementing compound, while remaining above that of the compound formed by interaction between the soluble collecting reagent and the salt or salts at the mineral surface; or it may be one that reduces the solubility of the slime particle below that of the possible cementing compound. If this hypothesis is correct, the reagent which inhibits slime-coatings must necessarily vary with the mineral that we are attempting to float and also with the chemical character of the slime particles.

In the case of calcite coatings on galena, several reagents are available. Any one should be satisfactory if it reacts with the galena surface to form thereon a compound which is less soluble than lead carbonate (cement) but more so than a lead compound of the collecting reagent; or one which reacts with the calcite surface to form thereon a compound which is less soluble than lead carbonate.

TABLE 1.—*Effect of Various Reagents*

Reagent	Solubilities, Mg. per Liter (9,10)		Coating
	Lead Salt	Calcium Salt	
None			Heavy
Potassium chromate	0.1	Very soluble	Very little
Sodium carbonate	1.7	65	Little
Sodium phosphate	0.14	23	None
Sodium oxalate	1.5	6.7	Little
Sodium tungstate	Ins.	2	Little
Water glass	Ins.	95	None

Several reagents have been tested, with the results shown in Table 1. Cleavage surfaces and polished surfaces of galena were treated for 5 min. in a pulp composed of 50 grams of −200-mesh calcite suspended in 200 c.c. of a solution containing one part of reagent to 10,000 parts of water. These data show that all lead salts of the acid ions supplied by the stated reagents are less soluble than lead sulfate, which is present at the surface of galena; hence a reaction between lead sulfate and the reagent is indicated, forming the less soluble lead salt at the surface of galena.

Similarly, the solubilities of the calcium salts of the acid ions supplied by the reagents also indicate a reaction between calcium carbonate and the reagent (except silicate and carbonate). But, all of these calcium salts are more soluble than the corresponding lead salts; hence, the lead salts will be formed in preference to the calcium salts. Finally, these lead salts are less soluble than lead carbonate which acts as the cement (excluding sodium carbonate); therefore, no reaction may be expected between calcite and a galena surface coated with these lead salts.

Water glass (sodium silicate) has been used for years as an addition reagent in flotation. Taggart(1) mentions it as suitable for inhibiting slime-coatings. As metallic silicates[10] are generally very insoluble,(15) water glass appears to be universally applicable.

11. A clean, polished surface of galena was exposed for 15 min. in an agitated pulp of −200-mesh calcite in distilled water (20 per cent solids). The particle was then removed, washed in three separate baths of distilled water, and dried. Fig. 8 shows the extensive slime-coating obtained. Another clean, polished surface of galena was similarly treated, but this time water glass (2 lb. per ton of solid) was added to the pulp. Fig. 9 shows the complete absence of slime-coating on the surface thus treated.

According to the chemical theory, the water glass forms a silicate or silicates of lead on the surface of galena, lead silicate being less soluble than lead sulphate; therefore the cement, lead carbonate, cannot be formed between galena and calcite because lead silicate is less soluble than lead carbonate.

The following experiments seem to confirm this hypothesis:

12. A 50-gram portion of −200-mesh galena was shaken with 300 c.c. of distilled water for 15 min., and filtered. The filtrate was analyzed for sulfate(11) and was found to contain 8.3 mg. of sulfate ion per liter of water.

13. Another 50-gram portion of −200-mesh galena was shaken for 15 min. in 300 c.c. of a solution containing 26 mg. of silicic acid per liter of water,(16) and filtered. The filtrate was divided into two parts. One part was found to contain 17.8 mg. of sulfate ion per liter of water. The other part was analyzed for silicic acid, and it was found that the galena had abstracted one-half of the available silicic acid.

Presumably, the reaction that had taken place was:

$$PbSO_4 + \text{Silicate ions} \rightarrow \text{Lead silicate} + SO_4^{--}$$

14. The same galena, after having abstracted the silicate ions, was shaken for 15 min. with 300 c.c. of a solution containing 25 mg. of potas-

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[10] Water glass added to solutions of metal salts forms metallic silicates or, in some cases, double silicates.(15)

sium ethyl xanthate per liter of water, and filtered. Tests of the filtrate showed substantially complete abstraction of xanthate ion[11] and that silicate ions had been thrown into solution.(16)

Apparently, the reaction had been:

$$\text{Lead silicate} + 2\text{KEtX} \rightarrow \text{Pb(EtX)}_2 + 2\text{Potassium silicate}$$

15. A 50-gram portion of −200-mesh calcite was shaken for 15 min. in 300 c.c. of a solution containing 26 mg. of silicic acid per liter of water, and filtered. Analysis of the filtrate showed that there had been no abstraction of silicate ions.(16) This was to be expected, as calcium carbonate is less soluble than calcium silicate.

The results of experiments 11 to 15 indicate that water glass acts as an inhibitor of calcite slime-coatings on galena by forming a very slightly soluble coat on the galena surface, which coating prevents reaction with the calcite but does not prevent reaction to form lead ethyl xanthate.

Magnitude of Slime-coatings

Looking at photomicrographs of slime-coated mineral surfaces, it is easy to underestimate the density of these coatings and thereby fail to recognize their importance.

Fig. 10 is a photomicrograph, at 900 diameters, of a polished galena surface exposed for 15 min. in an agitated pulp of −200-mesh calcite in distilled water (20 per cent solids). The diameter of the photomicrograph is 51 mm. and its area is 2040 sq. mm. In this field, the average number of particles inside a circle of 10 mm. diameter is 30, the area of that circle being 78.7 sq. mm.; whence the total number of particles in view is 780. But the diameter of the actual field on the galena surface is only 0.0568 mm. and its area, 0.0025 sq. mm.; hence, on the surface of galena there are about 320,000 particles per sq. millimeter.

The average dimensions of one of the slime particles in Fig. 10 are about 0.001 mm. by 0.001 mm., an area of 0.000001 sq. mm. Hence, in one square millimeter of mineral surface the total area occupied or "protected" by these particles is approximately 0.307 sq. mm., meaning that 31 per cent of the galena surface is substantially not galena but calcite.

The difficulty with which an air bubble may attach itself to such a slime-coated surface can be suggested by the following calculation: If we assume a molecule of lead ethyl xanthate, with which galena becomes coated when treated in potassium ethyl xanthate solution,(7) to be anchored to the mineral surface in such way that its total length is L, then we can roughly estimate L by adding the atomic diameters[17] of Pb (2.36Å), S (3.64Å), C (2.5Å), O (2.7Å), and 2(CH_2)[18](4Å), a total of 15.2Å. (1Å = 10^{-8} cm.)

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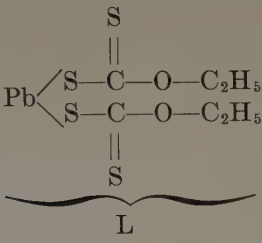
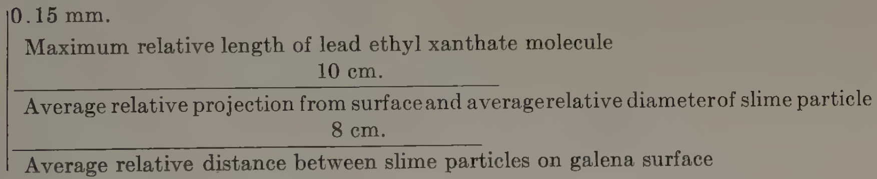
[11] Twenty-five c.c. of the original solution was put into a colorimetric tube and 25 c.c. of copper sulfate solution (100 mg. per liter) was added to it. An equal amount of the filtrate was similarly treated. The different amounts of cuprous ethyl xanthate precipitated in the two tests indicated abstraction of xanthate ion.



$$\underbrace{\mathrm{Pb}\begin{array}{l} \diagup\mathrm{S-\overset{\overset{S}{\|}}{C}-O-C_2H_5} \\ \diagdown\mathrm{S-\underset{\underset{S}{\|}}{C}-O-C_2H_5} \end{array}}_{L}$$

If, for convenience, we multiply the height of a slime particle (0.001 mm.), the distance between adjacent slime particles (0.0008 mm.), and L each by 100,000, we can plot these dimensions so as to show the enormous difference in the orders of magnitude of slime particles and lead ethyl xanthate molecules.


0.15 mm.
Maximum relative length of lead ethyl xanthate molecule
10 cm.
Average relative projection from surface and average relative diameter of slime particle
8 cm.
Average relative distance between slime particles on galena surface

Roughly, the condition is not unlike that of a grassy forest, the grass representing the xanthate molecules and the trees, the slime-coating particles, no inference being intended as to the shape of the particles.

SUMMARY

1. It is postulated that calcite slime-coatings on galena are due to metathesis between anchored lead ions on the galena and anchored carbonate ions on the calcite; this produces the almost insoluble lead carbonate which then acts as a cement between the galena surface and the slime particle.
2. It is also postulated that those reagents which inhibit calcite slime-coatings on galena operate by reacting with the sulphide mineral surface, or with the calcite surface, or with both, to form thereon compounds which are less soluble than lead carbonate.
3. Water glass (sodium silicate) is an excellent reagent for inhibiting slime-coatings on galena. It functions by forming relatively insoluble lead silicates on the surface of galena.
4. The magnitude of slime-coatings on galena is discussed.

ACKNOWLEDGMENT

Grateful acknowledgment is made to Prof. Arthur F. Taggart for the constructive criticism and inspiring encouragement given during the progress of this research.

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DISCUSSION

(*Arthur F. Taggart presiding*)

W. H. COGHILL,* Rolla, Mo.—Nothing has been said here about the means of grinding these ores. That has been our trouble for a number of years in the work on the flotation of nonsulfides. The reaction in the flotation cell when the crushing is done in a porcelain jar with flint pebbles is entirely different from that when it is done in an iron mill with iron balls. So in our work at Rolla we have standardized our work with flint-pebble grinding, and if we join any company in the flotation of tungsten or chromium or some other metal on which we have perfected flotation to the point where we are ready to help commercialize the work, we are so cranky about how the ores are to be ground that we would be afraid to start with an ordinary ball mill. We would urge that at least in the beginning of the work flint pebbles should be used. We know that a pebble mill has not much capacity for grinding, but with us it is a vital thing with the nonsulfides. Also with the sulfides on Platteville ores we find that the group of conditioning agents that is best if the ore is ground in an iron mill is not so satisfactory if the grinding is done in a pebble mill.

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* Metallurgist, U.S. Bureau of Mines.

V. E. Flanagan,* Dorchester, Mass.—Most of the grinding in a mill is done with steel balls in a steel mill. Why do you not run your laboratory work to parallel the work that is already being done in the concentrating plants and have results that you can put into practice without experimenting in commercial operations? It seems to me that would be a logical method of procedure.

W. H. Coghill.—It is not so vital after all with a company to switch over to a pebble mill. There is no reason why the ores generally should not be ground to ⅛ in. in crushers and rolls, and the part that remains to be ground down to flotation size is not a large percentage. Therefore it would not add a great deal to the cost of this operation to do the grinding with flint pebbles. The fact is, on some of the jobs the conditioning is so delicate that we cannot make the separation if we grind with iron balls.

R. H. Cromwell, New York, N.Y.—There is a possibility that slime-coating may explain the reason why, in some mills where fine grinding is known to be necessary to free interlocking minerals and gangue, the finer grind increases the losses in the tailing. The explanation would be that the finer grind produces more slime than the coarser grind, and that the larger amount of slime present increases the losses in the tailing by the slime-coating of the mineral particles.

A. F. Taggart,† New York, N.Y.—We have known that for several years. The question came up at a meeting two or three years ago at the time of the presentation of a paper by Mr. Fahrenwald. It is one of the reasons that we have been plugging away at the slime-coating problem for several years. We wanted to get at the method of control.

There is a rather interesting experimental fact, too, that we ran into at about that time, working with sulfide flotation. We were trying to see through the microscope what happened in the process of attaching bubbles to mineral particles. One of the experiments involved generating bubbles in the presence of deslimed ore, a sphalerite-silicate ore, by adding a small amount of calcium carbonate and then a small amount of acid to the pulp in a watch glass, and watching under the microscope the evolution of bubbles and their attachment to the sulfide particles. Sometimes the thing worked beautifully. Bubbles would grow on the sulfide particles shortly after the acid was added. But sometimes it did not work that way at all. We could not figure for a long time what was the matter, and since we were planning to use this experiment in court, we were distinctly nervous as to whether when we put it on it was going to be one of the ones that worked or one of the ones that did not work. Finally, we discovered that in the experiments that did not work there were certain points at which there was a very rapid evolution of bubbles. After quite a while we discovered that these points of evolution were distinctly magnetic, and then that they were iron, and that the particular pulps that gave this trouble had been ground on a bucking board, or else had been ground in a disk pulverizer tightened down hard. With that lead, we found that if we merely threw in a little iron filings we would not get our result, but if we ground in agate and did not put in the iron filings, we did get the result. That would seem to align exactly with Mr. Coghill's problem.

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It occurs to me, however, that that is a phenomenon that would arise in the agitation froth process, where we depend upon precipitation of the gas for adherence of the bubbles to the particles, and not in the pneumatic process, where we do not need such precipitation. I wonder if Mr. Coghill's experience would give anything on that. Was your flotation done in an agitation machine?

W. H. Coghill.—Mechanical agitation, all of it.

A. F. Taggart.—I do not think you would have that difficulty, if you used a pneumatic machine. Can you use a pneumatic machine for this flotation?

W. H. Coghill.—Are you able to use a pneumatic machine in the laboratory?

A. F. Taggart.—Yes, but it took me a long time to learn.

W. H. Coghill.—Probably we have not been as persistent as we should have been.

A. F. Taggart.—Mr. Sayre rigged up a little air-lift on a small Callow cell to return pulp from the lower end of the cell back into the feed end, and working that way and aerating slowly, taking time for the test, there is no trouble at all.

A. Legsdin,* New York, N.Y.—On page 399 Mr. del Giudice declares that the electrostatic charge theory was discarded completely and the problem was attacked from the standpoint of chemical reaction. What is the difference between the two? I cannot distinguish where the electrostatic picture ends and the chemical picture begins.

A. F. Taggart.—In so far as another person can relate what a third person is thinking about, I can tell you what I think Mr. del Giudice had in mind.

The electrostatic theory as we think of it in the laboratory involves the presence of charges on the mineral particles of like or different signs, and when those signs are unlike, it contemplates that the unlike charged particles, if brought closely enough together, will stick together by reason of electrostatic forces of attraction; where the signs are of like charge, they will be prevented from sticking together by reason of electrostatic forces of repulsion. The chemical theory grants that particles may be charged but thinks that when particles are charged, it is by reason of the presence at their surfaces of ionized salts of low solubility, and that when two particles with two different such ionized salts come together and adhere, they do so by reason of a reaction of metathetic type between the two different salts at the two different surfaces; specifically, for instance, in this particular case, between calcium carbonate and lead sulfate to form lead carbonate, and that the subsequent anchoring is due not to electrostatic attraction between a negatively charged calcium carbonate particle and a positively charged galena particle but to an actual cement composed of a number of molecules of lead carbonate that form at the contact surface of the two particles.

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* Mining and Metallurgical Engineer.

Production of High-grade Concentrate from Butte Copper Ores—Results of Laboratory Investigations

By Bayard S. Morrow* and George G. Griswold, Jr.,† Member A. I. M. E.

(New York Meeting, February, 1933)

The copper-bearing ores concentrated at the Anaconda plant of the Anaconda Copper Mining Co. are principally a mixture of copper and iron sulfides associated in a gangue consisting of quartz, lightly altered granite and feldspars, with sphalerite and galena and more rarely barite and hubnerite as accessory minerals. The predominant sulfide is pyrite.

The copper minerals present are: chalcocite, bornite (erubescite), enargite, chalcopyrite, covellite, tetrahedrite and some tennantite.

Normal concentrator practice removes a slime prior to ball-mill grinding. The deslimed ore is ground to approximately 10 per cent plus 65 mesh for flotation. Two selective flotation circuits are used, one for deslimed ore and one for slime.

Table 1.—*Metallurgical Results, Existing Flow Sheet*

Product	Weight, Per Cent	Assays, Per Cent				Copper Recovery, Per Cent
		Cu	Fe	SiO_2	Al_2O_3	
Ore	100.00	5.50				100.00
Mill concentrate	13.98	30.60	24.1	4.1	1.4	77.77
Slime concentrate	5.08	20.00	25.4	10.3	2.6	18.47
Total concentrate	19.06	27.77	24.5	5.75	1.7	96.24
Mill tailing	69.52	0.24				3.03
Slime tailing	11.42	0.35				0.73
Total tailing	80.94	0.256				3.76

Microscopic studies of concentrator products disclose such an interlocking of copper minerals with pyrite that any material increase in concentrate grade entails additional grinding unless recovery is sacrificed to a considerable extent.

During the latter part of 1931 and the early part of 1932 the problem of producing a high-grade concentrate (40 per cent Cu) with at least no

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diminution in recovery was extensively studied in the laboratory. The procedures evolved and the results obtained during the laboratory investigation are believed to be of interest in the art of metallurgy and it is with that thought in mind that this paper is presented.

The normal flow sheet for the production of high-grade concentrate is:

1. Produce a low-grade concentrate containing the bulk of the pyrite and a tailing containing a minimum of copper in a rougher circuit.

2. Regrind the low-grade concentrate and condition with pyrite depressants if necessary.

3. Float the reground, low-grade concentrate in a cleaner circuit, producing a final concentrate of acceptable grade and a cleaner tailing, which is returned to the original or rougher circuit.

This flow sheet was adopted for the initial laboratory tests. Preliminary tests clearly demonstrated the necessity for pyrite depressants in the cleaning circuit. Sodium cyanide and zinc sulfate were found satisfactory.

The usual laboratory flotation test is termed a single-stage test. It consists in the treatment of a single batch, usually 1000 grams, of ore and produces three products; that is, a concentrate, a middling and a tailing. The results obtained from single-stage tests are exemplified in Table 2.

Table 2.—*Results of Single-stage Tests*

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Heads	100.00	4.78	12.02		100.0	100.0
Final concentrate	7.22	37.3	15.2	9.0	56.3	8.4
Middling	39.70	5.1	28.5	29.8	42.3	86.8
Tailing	53.08	0.13	1.2		1.4	4.8

The rough concentrate that was reground, conditioned and refloated amounted to 46.92 per cent of the heads, assayed 10.03 per cent Cu and contained 95.2 per cent of the original copper.

Although the grade of concentrate is lower than expected and the middling weight and copper therein are too great, the scheme warranted further investigation.

A series of three-stage cycle tests were then made wherein the middling (cleaner tailing) from the first stage was added to the original head of the second stage, and the middling from the second stage was added to the original head of the third stage. One of the best stage tests is summarized in Table 3.

Here the real faults of a normal flotation flow sheet for the production of high-grade concentrate from Butte ores appear. The use of pyrite

TABLE 3.—*Three-stage Test*

Product	Weight, Per Cent	Assays, Per Cent		Recovery, Per Cent	
		Cu	Fe	Cu	Fe
Heads	100.00	4.81	13.3	100.0	100.0
Final concentrate	9.29	35.7	16.3	69.0	11.4
Final middling	13.44	8.5	25.8	23.7	26.1
Tailing: Stage 1	18.39	0.15	1.4	0.6	1.9
Stage 2	27.20	0.48	11.0	2.7	22.6
Stage 3	31.68	0.59	15.9	4.0	38.0

depressants, more powerful in action than high alkalinity with lime, are essential if concentrate grade is maintained near to 40 per cent copper. It is evident that the return of cyanide and zinc sulfate products to the original circuit via the cleaner tailing route has adversely affected recovery by depressing pyrite particles containing locked copper minerals.

To grind all of the heads to a fineness that would adequately free copper from pyrite not only presents an economic difficulty but also presents a problem of overgrinding.

At 10 per cent plus 65 mesh there is produced a considerable amount of free copper minerals. Further grinding at this point increases the amount of excessively fine free copper minerals and thereby aggravates losses.

The idea of two independent circuits was then conceived: (1) an open primary circuit, none of its products being returned to itself for retreatment; (2) a closed secondary circuit, the middling produced being returned to itself for retreatment. The objects in view were:

1. *Primary Circuit.*—To produce a high-grade concentrate, containing a goodly portion of the copper minerals freed by normal grinding; a low-grade concentrate or middling containing most of the pyrite and as much of the remaining copper as possible, and a tailing low in copper.

2. *Secondary Circuit.*—Regrind and condition the low-grade concentrate produced in the primary circuit and then subject the reground and conditioned low-grade concentrate to flotation, producing a secondary concentrate (high grade), a middling, to be returned to the head of the secondary flotation circuit, and a tailing containing so little copper that it may be rejected without further treatment. The main novelty in the scheme lies in the rejection of a secondary tailing that would, in all probability, assay well above the normal tailing regularly produced.

The scheme would avoid the faults of the original flow sheet (exemplified in Table 3) by: (1) removing, in the primary circuit, the bulk of the readily freed and readily floated copper minerals in the form of a high-grade product before an opportunity for excessive overgrinding presented itself; (2) confining the use of pyrite depressants to the secondary circuit, where they belong; *i. e.*, to a circuit where pyrite and copper minerals are adequately freed.

For convenience, deslimed ore was used as the heads for further laboratory work. Table No. 4 briefly sets forth the results obtained from single-stage tests. Concentrate grade is excellent, the middling contains but 17.4 per cent of the original copper as compared to 42.3 per cent for the rougher circuit (Table 2).

TABLE 4.—*Single-stage Tests*

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Head	100.00	5.18	14.2		100.0	100.0
Primary concentrate	4.94	45.3	12.7	5.1	43.2	4.4
Secondary concentrate	4.32	42.8	14.7	2.0	35.8	4.5
Total concentrate	9.26	44.2	13.7	3.7	79.0	8.9
Secondary middling	9.40	9.6	31.8		17.4	21.1
Primary tailing	60.21	0.19	4.5		2.1	19.1
Secondary tailing	21.13	0.40	34.1		1.5	50.9
Total tailing	81.34	0.23	12.2		3.6	70.0

The primary middling or heads to the secondary circuit was:

	Weight, Per Cent	Assay, Per Cent		Recovery, Per Cent	
		Cu	Fe	Cu	Fe
Primary middling	34.85	8.12	31.1	54.7	76.5

The metallurgy indicated in Table 2 was decidedly attractive and necessitated a series of cycle tests to determine the probable effect of the circulating middling in the secondary circuit. Laboratory experience in the treatment of Butte ores has established the five-stage cycle test as adequately exemplifying mill practice whenever the successive stage tailings remain practically constant in copper assay.

Since the ordinary 1000-gram laboratory machine would call for a series of 15 or more charges to secure enough primary middling for a five-stage cycle test in the secondary circuit, and since the primary circuit is entirely an open circuit, a laboratory ball mill and flotation machine capable of handling a 50-lb. charge was utilized for the primary circuit.

The primary middling thus produced was then thickened and divided into 1000-gram (dry weight) charges. Each of five 1000-gram charges was separately ground to −300 mesh with lime, sodium cyanide and zinc sulfate, and then transferred to the laboratory flotation cell, where it constituted the initial head for one stage of the cycle test for the secondary circuit.

Laboratory procedure for both the primary and secondary circuits is diagrammatically depicted in the accompanying flow sheet.

FLOW SHEET LABORATORY FLOTATION TEST

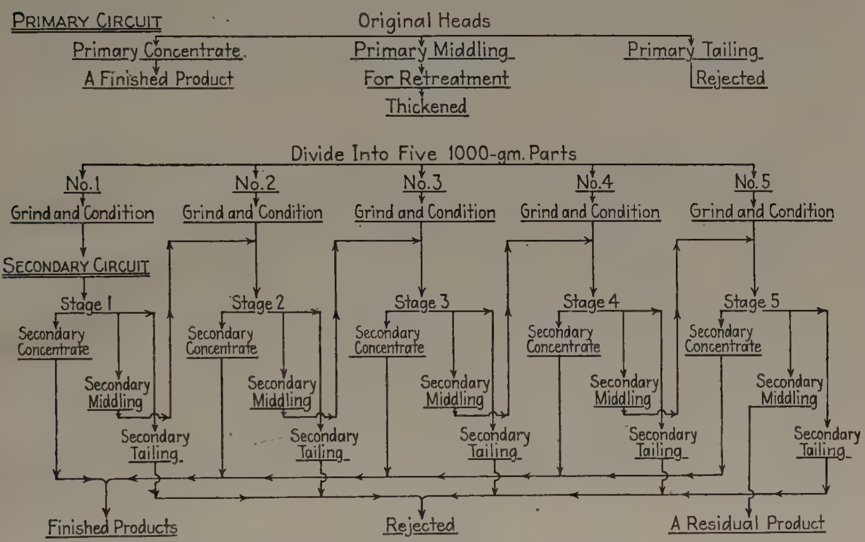

Table 5.—*Tests with Deslimed Ore*

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Heads	100.00	5.65	14.6		100.0	100.0
Primary concentrate	6.29	46.0	13.8	3.4	51.2	5.9
Secondary concentrate	6.15	36.4	18.3	5.0	39.6	7.7
Total concentrate	12.44	41.1	16.1	4.2	90.8	13.6
Secondary middling	6.66	5.6	35.8		6.5	16.3
Primary tailing	63.21	0.14	6.0		1.6	25.9
Secondary tailing	17.69	0.36	36.6		1.1	44.2
Total tailing	80.90	0.185	12.7		2.7	70 1

The five-stage cycle test results in the production of:

1. One primary concentrate from the original heads.
2. Five secondary concentrates from the primary middling.
3. One primary tailing from the original heads.

4. Five secondary tailings from the primary middlings.
5. One residual secondary middling from the primary middling.

The latter is the only unfinished product. For convenience the five secondary concentrates were combined for assay. Each individual secondary tailing is separately assayed in order that the effect of the circulating middling load may be truly gaged.

By following the procedure just described there was obtained results as depicted in Table 5.

Table 6 depicts the primary and secondary circuits viewed as separate entities.

TABLE 6.—*Primary and Secondary Circuits*

PRIMARY CIRCUIT

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Heads	100.00	5.65	14.6		100.0	100.0
Primary concentrate	6.29	46.0	13.8	3.4	51.2	5.9
Primary middling	30.50	8.75	32.7		47.2	68.2
Primary tailing	63.21	0.14	6.0		1.6	25.9

SECONDARY CIRCUIT

					Sec. Circ.		Total Ore	
					Cu	Fe	Cu	Fe
Heads (Primary middling)	100.00	8.75	32.8		100.0	100.0	47.2	68.2
Secondary concentrate	20.16	36.4	18.3	5.0	83.7	11.3	39.6	7.7
Secondary middling	21.84	5.6	35.8		13.9	23.9	6.5	16.3
Secondary tailing	58.00	0.36	36.6		2.4	64.8	1.1	44.2

The assays of the five secondary tailings were:

	Cu, Per Cent	Fe, Per Cent		Cu, Per Cent	Fe, Per Cent
No. 1	0.43	32.5	No. 4	0.35	37.2
No. 2	0.35	36.9	No. 5	0.35	37.6
No. 3	0.34	37.0			

An analysis of the results tabulated in Table 6 shows:

Primary Circuit.—The primary circuit recovers 51.2 per cent of the original copper in a concentrate assaying 46.0 per cent copper; loses but 1.6 per cent of the original copper as tailing and passes on to the secondary circuit 47.2 per cent of the original copper in the form of a low-grade concentrate or middling assaying 8.75 per cent copper and 32.7 per cent iron.

Secondary Circuit.—The secondary circuit recovers 83.7 per cent of the copper it treats, or 39.6 per cent of the original copper in concentrate assaying 36.4 per cent copper, and loses 2.4 per cent of the copper it treats, or 1.1 per cent of the original copper as a tailing assaying 0.36 per cent copper. There remains a residual or untreated middling amounting to 21.84 per cent of the weight of the heads to the secondary circuit assaying 5.6 per cent copper and containing 13.9 per cent of the copper treated in the secondary circuit, or 6.5 per cent of the original copper.

In mill practice the secondary middling would constantly be produced and constantly returned to the secondary circuit for retreatment and would, therefore, automatically distribute itself between secondary concentrate and tailing.

The almost constant copper assay of the individual tailings from the five laboratory stages clearly shows that the circulating middling has not increased the copper assay of the secondary tailings during the course of the test, and indicates that a circulating load of approximately constant copper assay has been built up.

The secondary circuit may then be summarized in Table 7, and the expected mill recovery for deslimed ore therefore becomes as shown in Table 8.

TABLE 7.—*Secondary Circuit, Middling Distributed*

Product	Weight, Per Cent		Assays, Per Cent			Recovery, Per Cent			
	Sec. Heads	Total Ore	Cu	Fe	Insol.	Cu	Fe	Cu	Fe
Heads	100.00	30.50	8.75	32.7		100.0	100.0	47.2	68.2
Secondary concentrate	23.28	7.10	36.4	18.3	5.0	96.8	13.0	45.7	8.9
Secondary tails	76.72	23.40	0.36	37.1		3.2	87.0	1.5	59.3

The application of the primary-secondary flow sheet to Anaconda slime presents new difficulties, chiefly surface or adsorptive effects of very fine gangue minerals. It is believed that the adverse effects of very fine minerals must be overcome by the use of adsorption or dispersion agents, in order that promotors, frothers, and chemical depressing or activating agents may react properly with the sulfide minerals.

TABLE 8.—*Deslimed Ore*

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Heads	100.00	5.65	14.6		100.0	100.0
Primary concentrate	6.29	46.0	13.8	3.4	51.2	5.9
Secondary concentrate	7.10	36.4	18.3	5.0	45.7	8.9
Total concentrate	13.39	40.91	16.2	4.2	96.9	14.8
Primary tailing	63.21	0.14	6.0		1.6	25.9
Secondary tailing	23.40	0.36	37.1		1.5	59.3
Total tailing	86.61	0.199	14.4		3.1	85.2

General adsorption and dispersion agents were tried. Among those giving fairly satisfactory results are calcium superphosphates, sodium silicate and aluminum sulfate.

The laboratory investigation for the treatment of slime was not carried to completion. Such results as have been secured are typified in Table 9. The dispersion agent used in the test work shown in this table was aluminum sulfate and the test depicts the results from a single-stage operation.

TABLE 9.—*Slimes*

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Head	100.00	5.63	8.6		100.0	100.0
Primary concentrate	7.62	36.2	14.4	12.1	49.0	12.8
Secondary concentrate	3.86	36.9	19.0	7.5	25.3	8.5
Total concentrate	11.48	36.4	15.9	10.5	74.3	21.3
Secondary middling	11.63	9.41	29.6		20.2	40.1
Primary tailing	64.21	0.34	2.2		1.6	21.7
Secondary tailing	12.68	0.73	14.7		3.9	16.9
Total tailing	76.89	0.40	4.3		5.5	38.6

The indicated possibilities for the treatment of Anaconda slime point to the production of a concentrate assaying 36.0 per cent copper and a tailing assaying from 0.40 to 0.45 per cent copper. The probable oxide copper assay of the tailing would be approximately 0.17 per cent, leaving 0.23 to 0.28 per cent as the sulfide assay.

Anaconda slime contains roughly one-half as much iron as does the deslimed ore. Therefore the elimination of pyrite is not so imperative in slime treatment as it is for deslimed ore. It is believed that the primary-secondary circuit can be advantageously modified for the treatment of Anaconda slime by so operating the primary circuit that a low-grade collective concentrate and primary tailing only are produced. The collective concentrate would then be ground and refloated in the secondary circuit producing a secondary concentrate and a secondary tailing of the approximate assays depicted in Table 9.

The expected metallurgy for such a flow sheet is shown in Table 10.

TABLE 10.—*Slime*

Product	Weight, Per Cent	Assays, Per Cent			Recovery, Per Cent	
		Cu	Fe	Insol.	Cu	Fe
Heads	100.00	5.9		7.2	10.000	100.00
Concentrate	15.31	36.4	14.4	12.0	94.44	30.61
Primary tailing	61.74	0.26			2.72	
Secondary tailing	22.95	0.73			2.84	
Total tailing	84.69	0.387			5.56	

The copper ores milled at the Anaconda plant during the period test work was being prosecuted averaged 5.5 per cent copper. The expected metallurgy for the treatment of ore of this grade is set forth in Table 11,

TABLE 11.—*Expected Metallurgy Primary-secondary Flow Sheet*

Product	Weight, Per Cent	Assays, Per Cent				Recovery, Per Cent
		Cu	Fe	SiO_2	Al_2O_3	Cu
Ore	100.00	5.5				100.00
Primary mill concentrate	4.83	46.0	13.8	2.6	0.8	40.43
Secondary mill concentrate	5.93	36.4	18.3	4.0	1.0	39.24
Secondary slime concentrate	2.52	36.4	14.4	10.0	1.0	16.71
Total concentrate	13.28	39.89	15.9	4.6	0.9	96.38
Primary mill tail	53.20	0.14				1.35
Secondary mill tail	19.54	0.36				1.28
Primary slime tail	10.19	0.26				0.49
Secondary slime tail	3.79	0.73				0.50
Total tailing	86.72	0.229				3.62

which shows the metallurgy actually obtaining in the plant during the period and reflects the existing flow sheet designed for the production of a moderately high-grade concentrate by selective flotation practice.

The goal set has been fully realized; *i. e.*, to produce a concentrate assaying approximately 40 per cent copper without any diminution in the percentage of copper recovered, as is evidenced by:

	PRIMARY-SECONDARY FLOW SHEET, PER CENT CU	PRESENT FLOW SHEET, PER CENT CU
Concentrate assay	39.89	27.77
Percentage of copper in concentrate	96.38	96.24

The flotation agents employed and their consumption in pounds per ton of original heads are assembled in Table 12.

TABLE 12.—*Flotation Agents Used*
(Pounds per Ton of Original Heads)

	Deslimed Ore			Slime		
	Primary Circuit	Secondary Circuit	Total	Primary Circuit	Secondary Circuit	Total
Lime	7.50	2.0	9.5	7.5	1.7	8.2
Sodium Aerofloat	0.10	0.03	0.13	0.10	0.03	0.13
Cresylic acid	0.12	0.06	0.18	0.24	0.08	0.31
S. D. pine oil	0.08	0.03	0.11	0.08	0.03	0.11
Coal-tar oil	0.06		0.06			
Sodium ethyl xanthate	0.06	0.08	0.14	0.20	0.07	0.27
Cyanide		0.10	0.10		0.13	0.13
Zinc sulfate		0.03	0.03			
Aluminum sulfate				1.0	0.3	1.3

The consumption of flotation agents is high. Additional laboratory work followed by a commercial campaign in the mill will undoubtedly point the way to some reduction. Lacking this, the consumption of flotation agents is listed as recorded in the laboratory.

MINERALOGICAL ANALYSIS OF CONCENTRATE

A microscopic count of representative laboratory concentrates indicates that a concentrate assaying 40 per cent copper would be made up as shown in Table 13.

The investigation carried on in the laboratory clearly indicates the fallacy of attempting to produce a high-grade concentrate from Butte ores by means of the usual rougher-cleaner flotation circuit. Unless all of the ore is ground fine enough to free the bulk of the copper, in this instance to minus 300 mesh, the cyanide and zinc sulfate products returned to the rougher circuit via the cleaner tailings depress pyrite particles containing

TABLE 13.—*Mineralogical Analysis of Concentrate*

	Weight, Per Cent	Distribution, Per Cent of Total								
		Cu	Fe	As	Sb	Zn	Pb	S	Insol.	Misc.
Bornite	26.0	36.4	25.8					23.4		
Chalcocite	13.7	27.4						9.0		
Chalcopyrite	14.9	13.1	27.6					16.6		
Covellite	0.8	1.2						1.0		
Enargite	17.3	21.1		100.0				17.9		
Tetrahedrite	0.7	0.8			100.0			0.6		
Sphalerite	3.2					100.0		3.5		
Galena	0.3						100.0	0.1		
Pyrite	16.3		46.6					27.9		
Gangue	4.6								100.0	
Not identified	2.2									100.0
Totals	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Calculated assay		39.8	16.3	3.3	0.2	2.1	0.2	31.2	4.6	2.3
Assay		39.7	16.7	3.3	0.19	2.18	0.17	30.7	4.6	

copper minerals. Economically speaking, minus 300 mesh grinding on all of the ore is not possible, and in addition the production of extremely fine particles from copper minerals freed at 10 per cent plus 65 mesh materially increases tailings losses.

The primary-secondary circuit as developed in the laboratory adequately overcomes the difficulties encountered in the rougher-cleaner circuit.

First, the bulk of the copper minerals freed by grinding to 65 mesh are removed in the primary float in the form of a high-grade concentrate.

Second, practically all of the copper minerals combined with pyrite are removed as a primary middling amounting to approximately one-third of the weight of the original ore.

Third, grinding to minus 300 mesh is limited to one-third of the weight of the ore and to a product in which the greater part of the copper minerals present are locked.

Fourth, the use of pyrite depressants is confined to the circuit in which they belong and they are not returned to a circuit where they can do harm.

As has been stated previously the only novelty proposed is the discarding of a secondary tailing relatively high in copper assay. The writers fail to recall any operating plants that have utilized this procedure. In this instance, at least, its use has made it possible to produce a concentrate assaying 40 per cent copper without any additional loss in recovery.

Microscopy in Flotation Research

By Guido R. M. del Giudice,* Member A.I.M.E.

A survey of the flotation literature of the past 10 years indicates an increasing use of the microscope as a tool for investigation. Thus, the metallurgical microscope has been used by Tucker and Head;[1] Taggart, Taylor and Ince;[2] Ince;[3] and del Giudice[4] to examine mineral surfaces that had been subjected to the action of various flotation reagents and to the action of slime pulps. It has also been used by Gaudin, Groh and Henderson;[5] Martin;[6] Morrow and Griswold;[7] and others to examine bakelite briquets of mill products. The microscope with transmitted light, both with bright-field and dark-field illuminations, has been used by Taggart, Taylor and Knoll[8] and by Ince[9] to observe Brownian movement and flocculation in flotation pulps. The use of the microscope at low-power magnifications with incident illumination for the examination of mill products has been known for years.[10] Finally, del Giudice[11] has used a relatively new type of illumination, the Ultropak, for the examination of slime-coatings.

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[1] E. L. Tucker and R. E. Head: Effect of Cyanogen Compounds on Floatability of Pure Sulfide Minerals. *Trans.* A.I.M.E. (1926) **73,** 354.

[2] A. F. Taggart, T. C. Taylor and C. R. Ince: Experiments with Flotation Reagents. *Trans.* A.I.M.E. (1930) **87,** 285.

[3] C. R. Ince: A Study of Differential Flotation. *Trans.* A.I.M.E. (1930) **87,** 261.

[4] G. R. M. del Giudice: A Study of Slime-coatings in Flotation. See page 398, this volume.

[5] A. M. Gaudin, J. O. Groh and H. B. Henderson: Effect of Particle Size on Flotation. A.I.M.E. *Tech. Pub.* 414 (1931).

[6] H. S. Martin: Microscopic Studies of Mill Products as an Aid to Operation at the Utah Copper Mills. *Trans.* A.I.M.E. (1930) **87,** 458.

[7] B. S. Morrow and G. G. Griswold, Jr.: Production of High-grade Concentrate from Butte Copper Ores—Results of Laboratory Investigations. See page 413, this volume.

[8] A. F. Taggart, T. C. Taylor and A. F. Knoll: Chemical Reactions in Flotation. *Trans.* A.I.M.E. (1930) **87,** 217.

[9] Reference of footnote 3.

[10] A. F. Taggart: Handbook of Ore Dressing, 1193, 1194. New York, 1927. John Wiley & Sons.

[11] Reference of footnote 4.

Optical, petrographic methods of ore examination are standard.[12–15]

It seems safe, therefore, to predict an increasing use of the microscope for the study of flotation phenomena in the future.

The fundamental equipment required and the optical accessories that must go with the microscope stand are expensive and the salesmen for the optical houses, willing and obliging as they are, have not the experience to advise intelligently on purchases. High magnification and exact resolution are needed, and a variety of types of illumination are made necessary by the difference in character of the specimens investigated. The investigator himself, therefore, must know the optical principles involved and the kinds of effects to be expected with the various optical tools available.

In addition to the use of the microscope for general examination of specimens, its application to the field of chemistry gives a rapid method of qualitative and quasiquantitative analysis which is not only expedient but sometimes the only way of attack open.[16]

Intelligent use of the microscope involves a certain amount of knowledge of the principles involved in the formation of the image and of the physical laws that govern the construction of the various optical systems which are part of the modern compound microscope stand. The choice of the best means of illumination and the proper use of microscope accessories are matters peculiar to the particular field of investigation and must be learned largely by experience in that field. The following presents an attempt to save the prospective user of these tools a great deal of time and money. It is a presentation of knowledge and experience which has taken the author several years of reading and actual work in the laboratory to attain.

In order to buy wisely, and use so that the greatest returns may be expected, it is felt that the following material, even though elementary in some parts, should be included in this development.

THE COMPOUND MICROSCOPE

The compound microscope, as the name implies, consists of two essential optical systems. Fig. 1 is a diagram showing the elemental functions of these two optical systems in the formation of the magnified

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[12] A. Johannsen: Manual of Petrographic Methods. New York, 1914. McGraw-Hill Book Co.

[13] L. McI. Luquer: Minerals in Rock Sections. New York, 1912. D. Van Nostrand Co.

[14] J. Murdoch: The Microscopical Determination of Opaque Minerals. New York, 1916. John Wiley & Sons.

[15] N. H. and A. N. Winchell: Elements of Optical Mineralogy. New York, 1909. D. Van Nostrand Co.

[16] E. M. Chamot and C. W. Mason: Handbook of Chemical Microscopy, 1 and 2. New York, 1930 and 1931. John Wiley & Sons.

image. The course of the rays (full lines) is constructed according to the laws of geometrical optics; the broken lines are merely construction lines. The *objective* or the first of the two optical systems forms a real, inverted image I' of the object O, which is placed somewhat beyond its principal focus f. The primary image I' is viewed through the second optical system, called the *eyepiece* or *ocular*, and really an ordinary magnifying glass, which forms a secondary, inverted, virtual image I'' at 10 in. (250 mm.) away from the eye of the observer. This distance has been set at 10 in. because the normal human eye can see objects best at that distance. Hence, this distance is called the *distance of distinct vision*. Optical parts of the compound microscope are therefore computed to form the final image at 10 in. from the eye of the observer. The distance between the back principal focal plane of the objective f' and the front principal focal plane of the eyepiece f'' is called the *optical tube length*. Objectives are computed to form a substantially optically perfect primary image at a definite place with respect to the front focal plane of the eyepiece; or, in other words, they are computed for a finite optical tube length. Unfortunately, each manufacturer has computed his objectives for an optical tube length that is different from that of other manufacturers. This is something to bear in mind when using different objectives. For convenience, the objective and the eyepiece are mounted at the opposite extremities of a tube which in turn is mounted on a stand, as in Fig. 2. In this figure, A is the top of the eyepiece and G the objective. The optical tube length is adjusted by regulating the *mechan-*

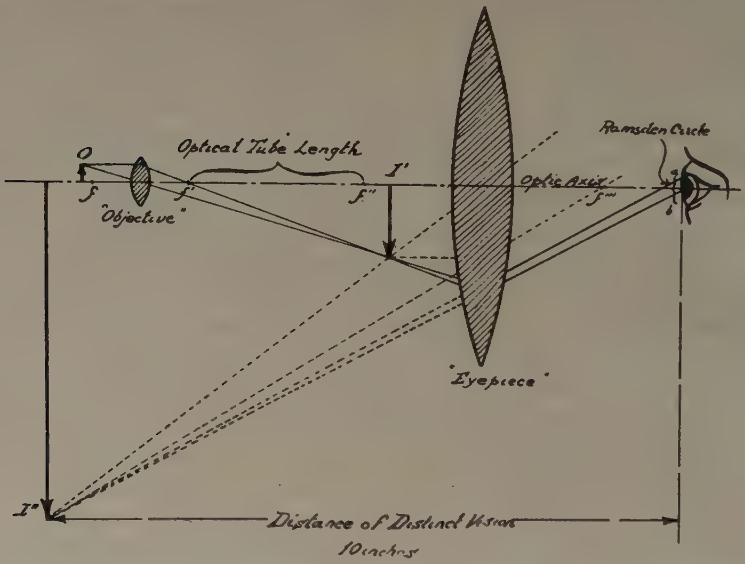

FIG. 1.—ELEMENTAL FUNCTIONS OF OPTICAL SYSTEMS OF THE MICROSCOPE. O, object; I', primary inverted image; I'', final image.

ical tube length T.L., which has a finite relationship with the optical tube length. This mechanical tube length can be increased or decreased by means of the *draw-tube B*. The entire *microscope tube C* can be lowered or raised by means of rack and pinion movement *D* (coarse adjustment); the final adjustment or focusing being performed with the fine adjustment screw *F*.

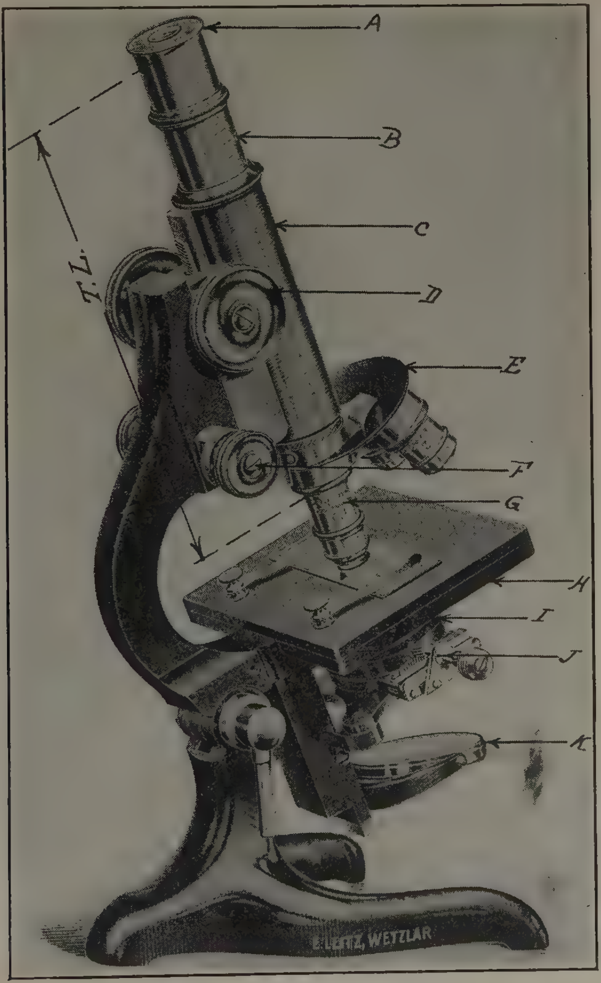

FIG. 2.—LEITZ MICROSCOPE.

At medium and high magnifications, a powerful concentration of light is required to illuminate the object under examination. For best results, the objective should receive light from an object placed at the apex of a similar cone of light as shown in Fig. 3. Theoretically, these conditions exist only when another objective is used which is identical in construction to the one that is part of the compound microscope. This illuminating objective, then, must in turn be focused on the object. Hence, the illuminating objective must be mounted on a rack and pinion mechanism. This is called the *Abbe substage*, *J* of Fig. 2. In addition to the rack and pinion mechanism, the Abbe substage is fitted with an iris diaphragm, which is used to control the size of the illuminating cone of light. This diaphragm is capable of side to side motion in order to give oblique illumination. (See page 443 and Fig. 23*B*.)

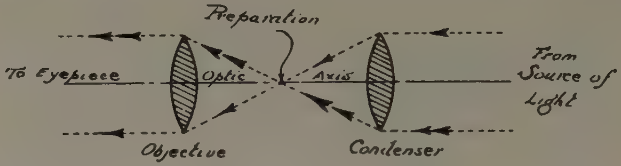

FIG. 3.—ARRANGEMENT OF ILLUMINATION FOR BEST RESULTS.

This involves considerable expense, because it implies possession of duplicate sets of objectives. This is done by some microscopists. However, with a view to economy, optical systems called *condensers*, *I* of Fig. 2, are manufactured, which are elastic enough, optically speaking, to be used in conjunction with the Abbe substage for a wide range of objectives. Thus condensers are essentially objectives that approximate the critical condition pictured in Fig. 3 for a number of objectives. When the preparation under examination is opaque, a *vertical illuminator* is employed. This uses the microscope objectives themselves for condensers, the light from the light-source being reflected down through the objective to the preparation and then back through the objective to the eyepiece (Figs. 25*A* and 25*B*). Thus, with vertical illuminators at least, the ideal condition pictured in Fig. 3 is realized.

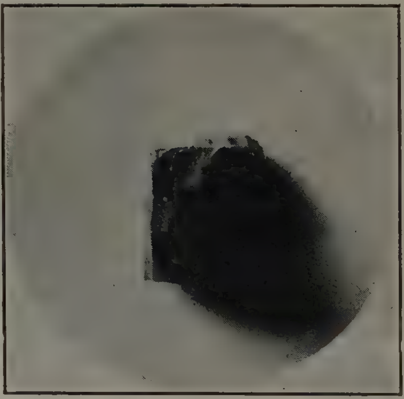

FIG. 4.—GALENA SURFACE. NATURAL SIZE.

MAGNIFICATION AND NUMERICAL APERTURE

Fig. 4 is a photograph of a galena surface taken at natural size, which microscopic examination has shown to be slime-coated. As in the photograph, visual examination does not disclose any slime-coating. If a slide is made of Fig. 4, and this slide is projected with a lantern on to a screen, the galena surface will appear several times larger, or it will be *magnified*. But still no slime-coating will be visible, while the microscope shows the slime-coating readily at comparable magnifications.

It seems, therefore, that magnification is not necessarily the only function of the microscope. The additional other function of the microscope is to resolve the slime-coating into individual particles of slime (Fig. 18). Therefore the microscope possesses *resolving power*; that is, the measure of the ability of an optical system to form an image of fine details of structure. In order to possess resolving power greater than the naked eye, the optical system must be able to receive and combine into an image a solid cone of light from the object which is larger than the cone of light received by the naked eye. The objective produces the primary image; therefore the resolving power of the microscope is vested in the objective. Fig. 5 shows a front lens of an objective receiving a cone of light from object point O; mO and $m'O$ represent the marginal rays of the cone of light. If the front lens could "take in" a cone of light whose marginal rays are represented by iO and $i'O$, the resolving power would be greater because the cone of light coming from O would be larger. Again, if the front lens could "take in" a cone of light whose marginal rays are represented by jO and $j'O$, the resolving power would be still greater. If the front lens is lowered to position X, it will "take in" a larger cone of light and its resolving power will be accordingly increased; and if lowered to position Y, the resolving power will be further increased. Hence, it may be expected that with a given diameter of lens, the shorter the focal length, the greater is the inherent resolving power of the lens. Also, at position X, the diameter of the lens can be reduced from mm' to $x'x''$ without any loss in resolving power because the cone of light "taken in" by the lens will be unchanged. Similarly at position Y, the diameter can be still further reduced without any loss in resolving power. In other words, as

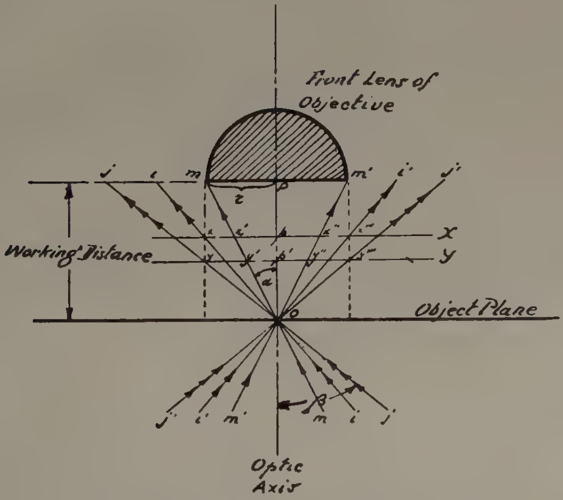

FIG. 5.—ILLUSTRATION OF RESOLVING POWER.

long as angle α is not changed, resolving power will not be changed. It appears, therefore, that, other things being equal, the apex angle of the solid cone of light, angle 2α, is the determining factor in the resolving power of a lens. The larger angle 2α becomes, the greater the resolving power, and conversely, the smaller angle 2α becomes, the smaller the resolving power. Angle α will be determined by the length of mO and the length r. Since angle mPO is a right angle, angle α may be expressed as the ratio r/Om or as the sine of angle α. Hence, resolving power is directly proportional to the sine of angle α. Further, since the rays entering an optically denser medium from a less dense medium are bent towards the normal to the interface between the two media, it may be possible, by placing a medium denser than air between the object plane and the front lens of the microscope objective, to cause all the rays included in the cone of light represented by marginal rays iO and $i'O$, and even the cone of light represented by marginal rays jO and $j'O$ to be "taken in" by the front lens of the objective. Thus, without increasing angle α, more rays of light from object point O will enter the objective. Therefore, the resolving power will be correspondingly increased. In other words, if rays jO and $j'O$ will enter the lens, although angle α does not change, the cone of light which the lens "takes in" will contain as many light-ray elements as the light cone whose apex angle is β. The higher the refractive index of the medium, the greater the number of rays that will be bent towards the normal PO, and therefore, the greater the number of rays that will be "taken in" by the lens. Hence, resolving power is directly proportional to the product of the refractive index of the medium intervening between object and front lens of objective, times the sine of angle α. Abbe called this product *numerical aperture*. Therefore, the resolving power of an objective is directly proportional to the numerical aperture of the objective. Fig. 5 shows that angle α may have a maximum value of 90°. Therefore, the maximum value that numerical aperture may have is equal to the refractive index times the sine of 90°, or the refractive index times 1. It appears quite clear that when angle α is 90°, it is impossible for the marginal rays to enter the objective because these rays are parallel to the object plane. Hence, the maximum, and it must be said, practical value of numerical aperture is equal to some value that is less than the refractive index of the medium used.

The method of increasing numerical aperture by using immersion liquids of higher refractive index soon reaches a limit. Fig. 6 shows the front lens of an objective focused on object point O at the lower surface of a cover glass. If the medium between the lower lens of the objective and the cover glass is air, then ray OA, which makes angle α with the optic axis, upon reaching the upper surface of the cover glass is refracted away from the normal and follows the course shown by AA'' and will not enter the lens ($\sphericalangle\gamma > \sphericalangle\alpha$). However, if a medium optically denser than air but not as optically dense as the glass of the cover glass is placed between the front lens of the objective and the cover glass, at A the ray OA will not be bent away from the normal as much as before ($\sphericalangle\beta < \sphericalangle\gamma$) and will just enter the front lens of the objective at A'. When such immersion fluid is used, there will be a critical value of angle α such as angle ω at which the ray (OBB') will not emerge. Rays such as ODD' will be reflected back into the cover glass. However, a ray such as OCC', which makes an angle ϵ with the optic axis, which is barely smaller than angle ω, will barely emerge. If such a ray is to enter the front lens of the objective, it then becomes necessary that the front lens of the objective be in substantially direct contact with the cover glass. Other things being equal, such a condition then represents the maximum, limiting, realizable use of numerical aperture that can be obtained with this medium as immersion fluid. In order to increase the numerical aperture if the lens is to "take in" a ray such as OE, which makes an angle with the optic axis slightly larger than angle ϵ, its diameter must be larger.

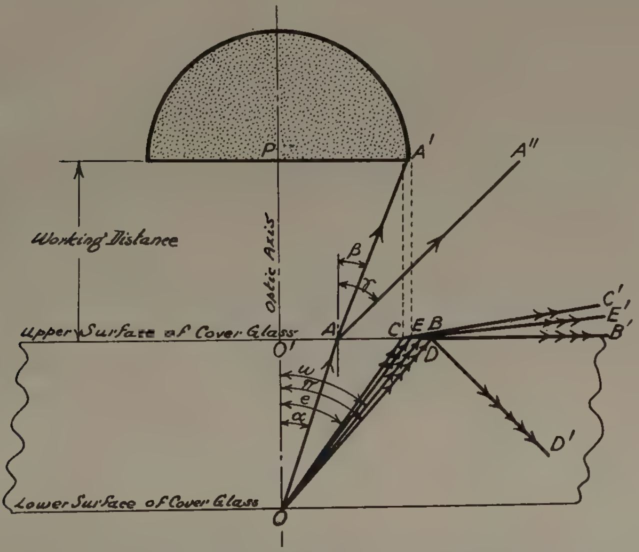

FIG. 6.—COURSE OF LIGHT RAY A WHEN MEDIUM BETWEEN FRONT LENS OF OBJECTIVE AND PLANE OF OBJECT IS AIR.

From the foregoing, it seems clear that with a given focal length the only way of increasing numerical aperture is to increase the diameter of a lens. However, since the front lens of a microscope objective (see page 436) has a diameter that is fixed by the diameter of the other component lenses that make up the microscope objective, and also since the back lens of the objective is the largest and its diameter in turn is fixed by the diameter of the metal mount of the objective, lenses of a given focal length have a limiting numerical aperture. Lenses that have a high initial magnification usually possess a short focal length. Since short focal length makes possible (Fig. 5) a higher numerical aperture without increasing the diameter of the lens, it is found that lenses with a high initial magnification also have a relatively high numerical aperture.

The immersion fluids used are:

Medium	Refractive Index	Maximum Num. Ap.
Air	1.0	0.95
Water	1.33	1.25
Glycerine	1.46	1.25
Cedar-wood oil	1.515	1.42
Alpha-mono-brom-naphthalene	1.658	1.60

The theory of microscopic vision postulates that images formed by the microscope are in part the result of diffraction effects. Since diffraction effects are dependent upon the wave length of the light employed and the angle at which light impinges upon an object, it has been shown that for a narrow axial cone of light, the dimension of the smallest structure that can be seen with an optical system or *limit of resolution* is the quotient of the wave length of the light used divided by the numerical aperture of the objective employed. If the object is illuminated by a narrow, oblique pencil of light (oblique and dark-field illumination, see Figs. 23*B* and 23*C*) and if the oblique pencil includes the most extreme, marginal rays that can pass through the objective, the limit of resolution is smaller than before (or the resolving power is greater) and is the quotient of the wave length of light employed divided by twice the numerical aperture of the objective used. Thus, by decreasing the wave length of the light employed, it is possible to increase the resolving power. Ultraviolet light (light of very short wave length) has been used. However, the cost of the apparatus (optical systems must be made of quartz because optical glass is substantially opaque to ultraviolet light), and the rather difficult technic involved (the human eye cannot see ultraviolet light), make this a tool valuable only to the experienced microscopist.

Thus, the microscope objective supplies a primary image in which presumably all the fine details of the structure in the object appear. The function of the eyepiece is to supply additional magnification of this primary image to make all available detail present therein visible to the eye of the observer. Without the proper amount of magnification, these details, although present in the primary image, will not be visible. The question arises: How much must this primary image be magnified? The following example shows how the total magnification to be used with a given objective is arrived at.

The average wave length of white light (white light is made up of light of different wave lengths) is roughly 0.0005 mm. Using white light, an objective with a numerical aperture of 0.95 has a limit of resolution of $W.L./2N.A. = 0.0005/2 \times 0.95 = 0.0005/1.90 = 0.000263$ mm. This means that this objective can resolve detail that is separated by 0.000263 mm. The objective has an initial magnification of 40 diameters. Hence, in the primary image produced by this objective, detail will appear separated by $40 \times 0.000263 = 0.01052$ mm. The normal human eye at 250 mm., or at the distance of distinct vision, can see detail that is separated by 0.11 mm., and no less. Therefore, in order to make detail separated by 0.01052 mm. visible to the eye, it becomes necessary to magnify the primary image, and this is done by the eyepiece. The additional magnification required will then be $0.11/0.01052 = 10$ or 10×. Thus, the total magnification employed should be 40 × 10, or 400. This is equivalent to 421 × 0.95, or about 400 times the numerical aperture of the objective. The number 400 is called the *visibility factor.* Thus, visibility factor is the number which when multiplied by the numerical aperture of a microscope objective will give the total magnification (that of the objective times that of the eyepiece) at which all details may be comfortably seen in the final image.

A number of similar calculations for different objectives will always give about the same visibility factor. Experience has shown that a visibility factor of 400 is really too small to comfortably see all details. Depending on the sight of different individuals, visibility factors between 600 and 900, say 800, may be used. Above 1000 the details of the primary image will be so magnified that they will not appear sharp. Thus, if the detail should correctly appear as a point, with a high visibility factor it will appear as a circle. This circle is referred to as the *circle of confusion.* The smaller the circle of confusion, the sharper the point.

If an objective has a numerical aperture of 0.95, and an initial magnification of 40×, the total magnification should not be more than 760×. Therefore, an eyepiece with a component magnification of 15× should be used giving a total magnification of 600×. The maximum magnification permissible with an objective of 0.95 numerical aperture can be approximated in photography by extending the camera beyond 250 mm. If a total magnification of 600× is attained with an objective whose numerical aperture is 0.30, so-called "empty magnification" is obtained. The result is no better than that obtained by projecting an image at much lower magnification on to a screen by means of a lantern. The higher magnification will not show any more detail than that which existed in the original image at lower magnification. The highest total magnification that should be reached with an objective whose numerical aperture is 0.30 is 240×. Fig. 7 is a photomicrograph of a calcite slime-coating on a polished galena surface taken at 600× with an objective having a numerical aperture of 0.30. Fig. 8 is a photomicrograph of the same at identical magnification, but an objective with a numerical aperture of 0.95 was used. The definition obtained in Fig. 8 is so clear when compared with that of Fig. 7 that no explanation seems necessary.

Therefore, in objectives, the numerical aperture is the important thing, not the magnification.

The best cedarwood oil immersion objective manufactured today, as stated, has a numerical aperture of 1.40. It seems therefore that with ordinary light a maximum magnification of about 1400× is all that can be reached. Beyond that, nothing can be seen that the magnification of 1400× does not disclose.

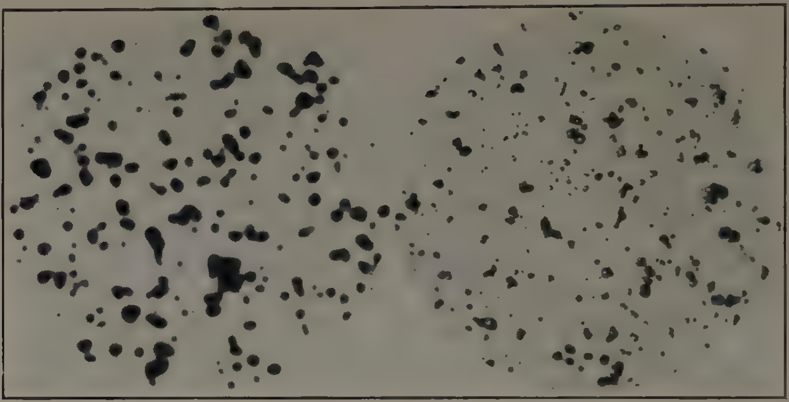

FIG. 7. FIG. 8.

FIG. 7.—CALCITE SLIME-COATING ON POLISHED GALENA SURFACE. N.A. 0.30. × 600.
FIG. 8.—SAME SAMPLE AS IN FIG. 7, BUT WITH N.A. 0.95. × 600.

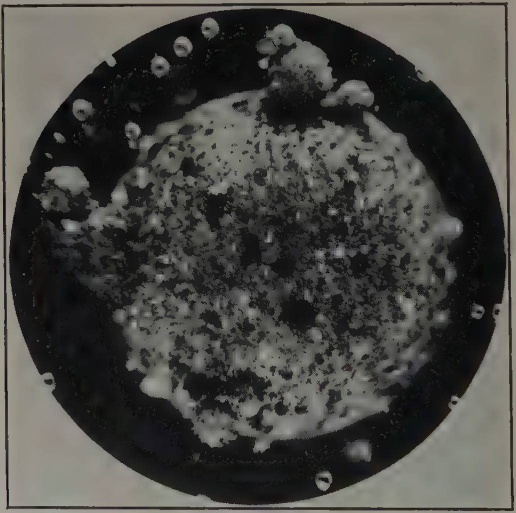

FIG. 9.—MINERAL-COATED AIR BUBBLE. N.A. 0.08. × 30.

Up to this point the impression created has been one that indicates the use of the highest available numerical aperture for a given magnification. However, for some observations, numerical aperture is not the deciding factor. An illustration of this fact is shown by Figs. 9 and 10. Fig. 9 is a photomicrograph of a mineralized air bubble taken at 30 diameters. The object of this figure was to bring into reasonable focus as much of the curved surface of the mineralized bubble as was possible without sacrificing definition to any great degree. An objective with a numerical aperture of 0.08 was chosen. Fig. 10 is a photomicrograph of another mineralized air bubble taken also at 30 diameters, but the object of this figure was to show the water globules (arrows) on the surface of the mineral particles in the air-water interface. In this case that surface was the important thing. An objective with a numerical aperture of 0.15 was used. The difference between the objects sought in Figs. 9 and 10 was a matter of *depth of focus*. The objective of lower numerical aperture possessed a greater depth of focus than the objective of higher numerical aperture. In other words, depth of focus varies inversely as the numerical aperture. This is a fact which the amateur photographer often unconsciously takes into consideration when he "closes down" the iris diaphragm of his photographic lens in order to bring "more of his subject" into "sharp focus."

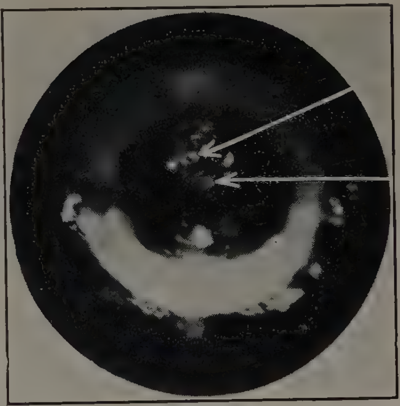

FIG. 10.—GALENA PARTICLES ADHERING TO AIR BUBBLE. N.A. 0.15. × 30.

Fig. 11 shows two lenses of different numerical aperture. In A, points P and Q when observed through the microscope will appear as circles with diameters xx' and yy', respectively. If xx' and yy' initially are of such dimensions that when magnified through the microscope they come to the eye of the observer just equal to 0.11 mm., the circles will appear substantially as sharp points. Although the focus is at O, all points between P and Q will appear as sharp points, their circles of confusion being smaller than those of points P and Q. Therefore the lens in A has a depth of focus equal to PQ. In B, although aa' and bb', the circles of confusion for points P' and Q' respectively, are at the same distance away from O' as xx' and yy' are from O in A, they are larger than xx' and yy'. Therefore P' and Q' will not appear to the observer as sharp points. Only at n' and m', at a distance away from the focus that corresponds to that of aa' and bb' of A, do we find the same diameter as that of xx' and yy' of A. Therefore, the lens in B, which has a higher numerical aperture than the lens in A, has a depth of focus equal to $n'm'$, distinctly shorter than the depth of focus of the lens in A.

By means of the aperture iris diaphragm in the Abbe substage, Figs. 2*J* and 23*A*, and the aperture iris diaphragm in the vertical illuminator, Fig. 25*A*, it is possible to reduce the numerical aperture of a given objective almost at will in order to increase the depth of focus. This reduction can be carried too far, to the point that definition is almost entirely lost. Fig. 12 is a photomicrograph similar to Fig. 8, but with the aperture diaphragm too much closed. The definition, by comparison, is almost entirely lost. The specks appear triangular and have lost their original shape. A good rule to follow is: Never close the aperture diaphragm so much that less than two-thirds of the back lens of the objective (observed by removing the eyepiece and looking down the tube of the microscope) is illuminated. If greater depth of focus is desired, it is better to employ another objective of lower numerical aperture rather than to risk the chance of obtaining false images by excessive reduction of aperture with the aperture diaphragm.

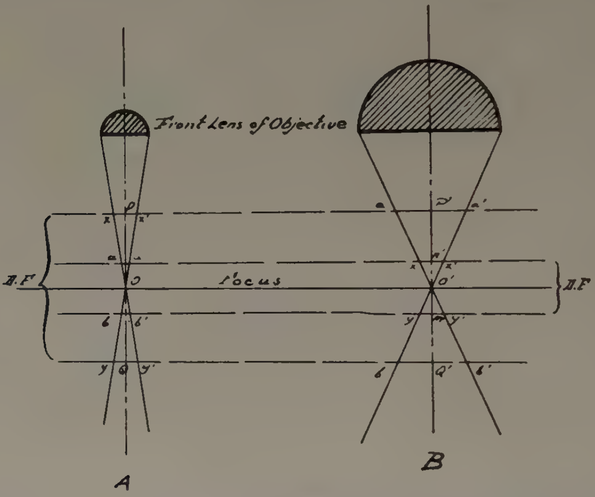

FIG. 11.—DEPTH OF FOCUS OF TWO LENSES OF DIFFERENT NUMERICAL APERTURE.

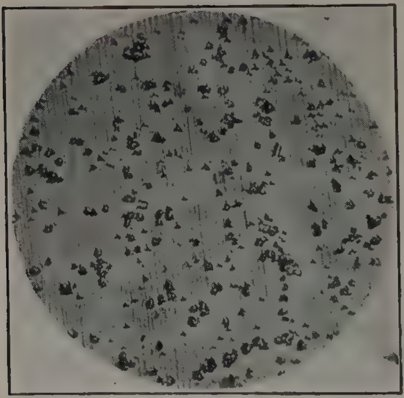

FIG. 12.—PHOTOGRAPH SIMILAR TO FIG. 8, BUT WITH APERATURE DIAPHRAGM CLOSED TOO FAR. × 600.

OBJECTIVES

Microscope objectives are not single lenses, as shown in Fig. 1. Lenses have inherent optical defects, which must be corrected if a substantially perfect primary image is to be expected. In order to correct these defects, other lenses must be added to the optical system. Therefore, the microscope objective is really an optical system made up of several lenses. The optical defects of a lens that are corrected by the addition of other lenses are *spherical aberration* and *chromatic aberration.*

Rays that pass through the marginal elements of a lens come to a focus different from that of the rays that pass through the central elements of the lens (Fig. 13*A*). The latter come to focus at the same place as the principal focus. The greater the angle made by the ray with the optic axis of the lens, the greater the *spherical aberration.* One method of correcting this defect is to reduce the aperture of the lens, by means of a diaphragm, so as to eliminate the marginal rays. This method has the drawback of also reducing the resolving power. The other method used in practice is to use an additional lens made of a glass of different refractive index from that of the glass of the first lens, as in Fig. 13*B*. Proper choice of glass, and judicious computation of the thickness and shape of this additional lens, will cause the marginal rays to come to substantially the same focus as the central rays.

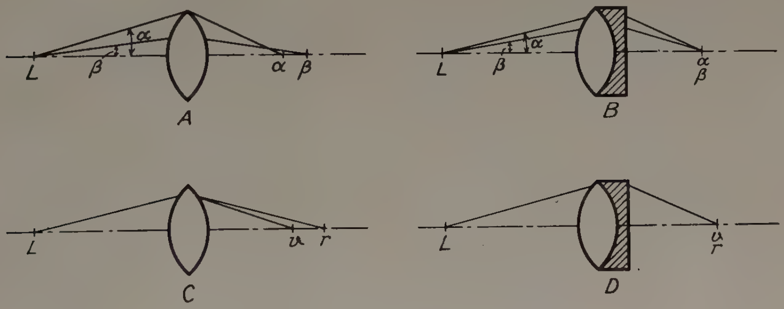

Fig. 13.—Examples of aberrations.

Since lenses are substantially prisms, rays of white light that pass through the marginal elements of a lens are dispersed into rays of different colors (different wave lengths). The rays of shorter wave length come to a focus at a point nearer the lens, and the rays of longer wave length come to a focus farther away from the lens (Fig. 13*C*), causing *chromatic aberration.* One method of correcting this defect is again to reduce the aperture of the lens, thereby allowing only the central rays to come through, which do not suffer much from chromatic aberration. This method, of course, is objectionable. The other method is to use additional lenses as shown in Fig. 13*D*. However, as will be presently shown, one additional lens does not remove chromatic aberration as efficiently as one lens removes spherical aberration, and in practice several lenses may be used, depending on the degree of correction desired.

Optical glasses, unfortunately, do not possess the property of dispersing light equivalent amounts for different wave lengths. An example will show why it is necessary to use more than one lens to correct chromatic aberation. A certain glass *A*, of a given refractive index, disperses white light so that on an arbitrary scale the difference between red, *R*, and yellow, *Y*, is 15 units; the difference between yellow, *Y*, and green, *G*, is 50 units, and the difference between green, *G*, and blue, *B*, is 35 units. Another glass B, of a different refractive index gives: *R* to *Y*, 10 units; *Y* to *G*, 55 units; *G* to *B*, 40 units.

If a chromatic combination is made from these two glasses, Fig. 14 will show the degree of chromatic correction obtained. Thus, *R* and *G* will be corrected but *Y* and *B* will not.

Another glass, *C*, will give: *R* to *Y*, 15 units; *Y* to *G*, 50 units; *G* to *B*, 38 units. And still another glass, *D*, will give: *R* to *Y*, 15 units; *Y* to *G*, 50 units; *G* to *B*, 36 units.

If a chromatic combination is made from these two glasses, Fig. 15 will show the degree of correction obtained. Since in combination *AB* the *B* was overcorrected, and since in combination *CD* the *B* is undercorrected, a combination of *AB* and *CD* should substantially correct *B*. For correcting *Y* in combination *AB*, it becomes apparent, then, that still another combination must be used.

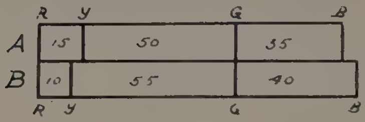

FIG. 14.

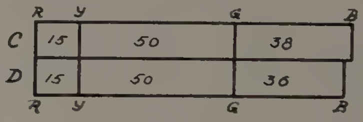

FIG. 15.

FIG. 14.—DEGREE OF CHROMATIC ABERRATION IN COMBINATION OF GLASSES *A* AND *B*.
FIG. 15.—DEGREE OF CHROMATIC ABERRATION IN COMBINATION OF GLASSES *C* AND *D*.

Elimination of aberration is the goal of every optical computer. Different optical glasses and even fluorite are used to approximate the ideal conditions. It must be borne in mind that while eliminating one aberration, say chromatic aberration, the other, spherical aberration, must also be eliminated, while at the same time the objective must be computed for a given magnification, a given tube length, and a given focal length. The problem is indeed very complicated.

Depending on the degree of optical correction they possess, objectives are divided into two general classes; viz., achromatic objectives and apochromatic objectives.

Achromatic objectives are the cheaper and are corrected for two colors in chromatic aberration and one color in spherical aberration. This color is olive-green and was so chosen because the human eye is most sensitive to it. It is therefore necessary to use a green filter with these objectives in order to obtain the best results. Fig. 16 is a photomicrograph of calcite slime-coating on galena taken with an achromatic objective of numerical aperture equal to 0.85 at 600 diameters (well within the range of 800 × N.A.) but without a green filter. Notice the relative lack of sharpness of detail when compared with Fig. 8.

Apochromatic objectives are expensive and are corrected for three colors in chromatic aberration and two colors in spherical aberration. Only a faint tertiary spectrum remains. With high-power apochromatic objectives there is a residual error, which can be eliminated (compensated) by introducing purposely an error in the eyepiece. These eyepieces are called *compensating eyepieces* and should always be used with apochromatic objectives. Fig. 17 is a photomicrograph similar to Fig. 8. The same apochromatic objective was used, but a noncompensating eyepiece was used with it. The photomicrograph is of such poor quality that it is self-explanatory. With apochromatic objectives, because of their optical construction, a higher numerical aperture is realizable for a given

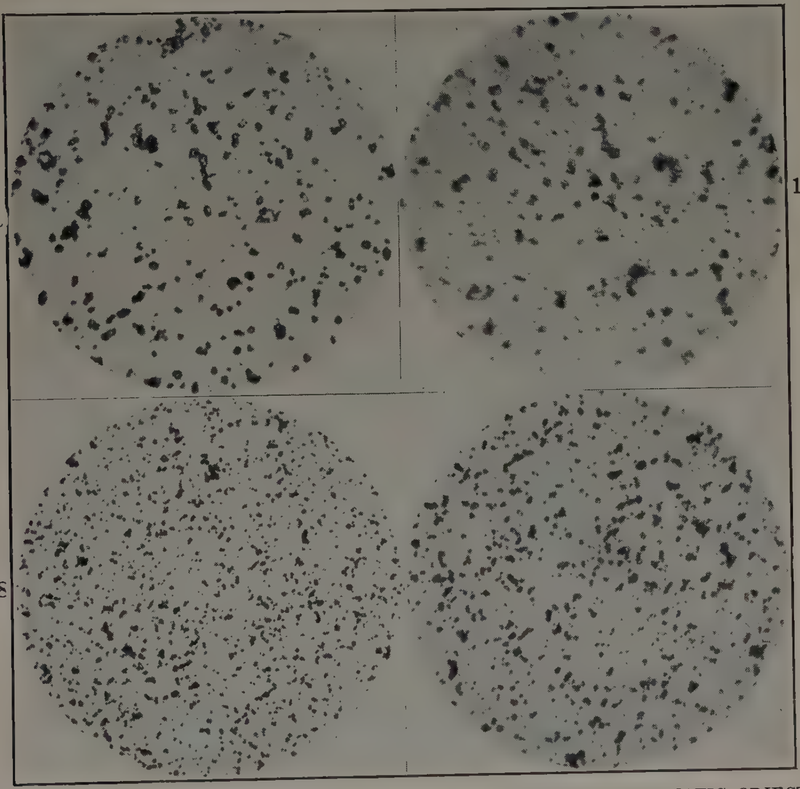

FIG. 16.—CALCITE SLIME-COATING ON GALENA TAKEN WITH ACHROMATIC OBJECTIVE WITHOUT GREEN FILTER. × 600.

FIG. 17.—PHOTOGRAPH SIMILAR TO FIG. 8, BUT TAKEN WITH APOCHROMATIC OBJECTIVE WITH NONCOMPENSATING EYEPIECE. × 600.

FIG. 18.—CALCITE SLIME-COATING ON GALENA TAKEN WITH APOCHROMATIC OBJECTIVE. N.A. 065. × 300.

FIG. 19.—SPECIMEN SIMILAR TO THAT IN FIG. 18, TAKEN WITH ACHROMATIC OBJECTIVE. N.A. 0.40. × 300.

focal length than for achromatic objectives. Therefore, with an apochromatic objective of a given focal length, greater resolving power may be expected than that obtained with an achromatic objective of identical component magnification. Fig. 18 is a photomicrograph of a calcite slime-coating on galena at 300×, taken with an apochromatic objective with an initial magnification of 22× and a numerical aperture of 0.65. The details of Fig. 18 appear considerably sharper than those shown by Fig. 19, which is a photomicrograph of a similar specimen taken at 300× with an achromatic objective having a component magnification of 20× and a numerical aperture of 0.40.

Each one of these classes of objectives is subdivided into *standard-mount* and *short-mount* objectives. The standard-mount objectives are used with transmitted light and are corrected for a given thickness of cover glass. At medium and high powers, if a cover glass is used which has a thickness different from that for which the objective was computed, an error in spherical aberration will be introduced. As was shown in Fig. 13*A* and on page 437, spherical aberration depends upon the angle made by the entering ray with the optic axis of the lens. In Fig. 20, with a cover-glass thickness shown by *XZ*, ray *A* is the limiting ray the lens can "take in." Ray *A* is refracted at *n* when it passes from the glass of the cover glass into air and just enters the lens at *Q*. Thus, for cover-glass thickness *XZ*, the lens must correct for the spherical aberration of a ray that makes angle β with the optic axis. However, if the cover-glass thickness is increased so that it becomes *YZ*, *A* will no longer be the limiting ray; ray *B*, which before did not enter the lens, will follow the

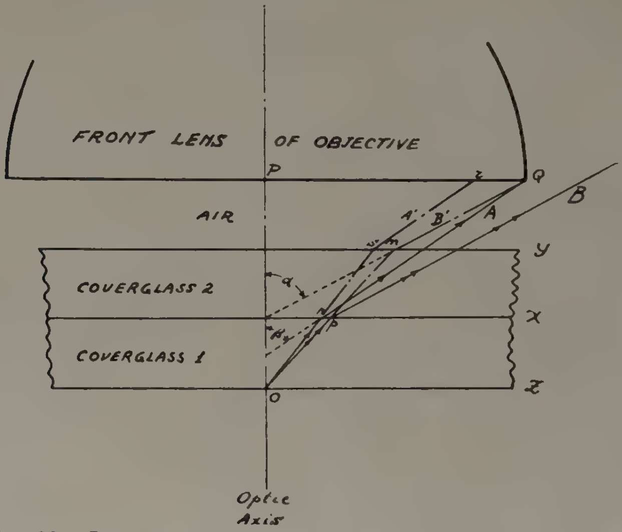

FIG. 20.—ILLUSTRATION OF EFFECT OF COVER GLASS THICKNESS.

course of ray B' and will become the limiting ray. This light ray makes angle α with the optic axis. Now, if the lens is corrected for cover-glass thickness XZ with an accompanying spherical aberration caused by rays whose maximum angle is β, this lens will not correct the spherical aberration introduced by a ray that makes a greater angle with the optic axis. (Angle α is greater than angle β.) Therefore this lens will have a residual error in spherical aberration introduced by the greater thickness of the cover glass. Thus, departures from the cover-glass thickness for which the lens is computed will introduce errors in spherical aberration. Good medium-power and high-power objectives are fitted with *cover glass correction collars,* which, by reducing or increasing the distance between component lenses within the objective mount, enable the user to correct at will for departures from the correct thickness of cover glass. The error is almost negligible for lenses of lower numerical aperture because the apex angle of the cone of light entering the objective is relatively small and therefore the spherical aberration is almost negligible (see page 437).

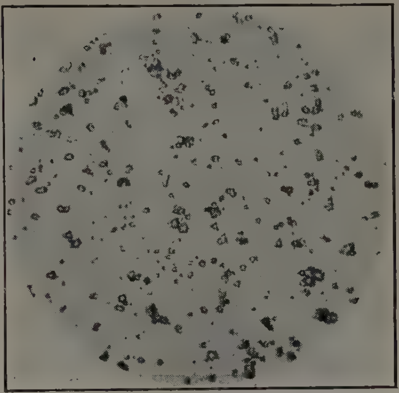

FIG. 21.—PHOTOGRAPH SIMILAR TO FIG. 8, BUT WITH TUBE LENGTH SET AT 20 MM. LESS THE CORRECT LENGTH. × 600.

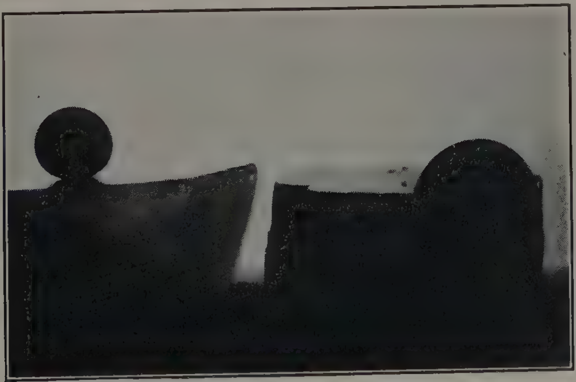

FIG. 22.—GLOBULES OF COAL-TAR CREOSOTE ON CLEAN SURFACES OF CALCIT AND GALENA IN WATER. × 5.

The short-mount objectives are used with vertical illuminators (see page 443) and are corrected for no cover glass.

As stated above, objectives are computed for a given tube length, and if good results are to be obtained the tube length for which a given objective is computed must be strictly adhered to. Fig. 21 is a photomicrograph similar to Fig. 8, but taken with a tube length set at 20 mm. less than the correct length. The relatively poor quality of the photomicrograph can be observed. Each manufacturer of microscope objectives computes the latter for different tube lengths. Therefore, when using objectives from various sources, care should be taken to adjust the draw-tube to the setting required by a given objective.

In addition to the compound microscope, photographic lenses of short focal length such as *microsummars* and *microplanars* are very useful in the study of contact angles. Fig. 22 is a photomicrograph of the behavior of coal-tar creosote on polished, clean surfaces of calcite and galena in water. These lenses are highly corrected optically and have a wide field of application.

EYEPIECES OR OCULARS

Microscope eyepieces consist of two lenses mounted at the opposite ends of a tube. The lens nearest the eye is called the *eye lens*, and it does the actual magnifying. The other lens is called the *field lens*, and simply changes the course of the rays which come from the objective so that the primary image may be of dimensions that come within the range of the eye lens.

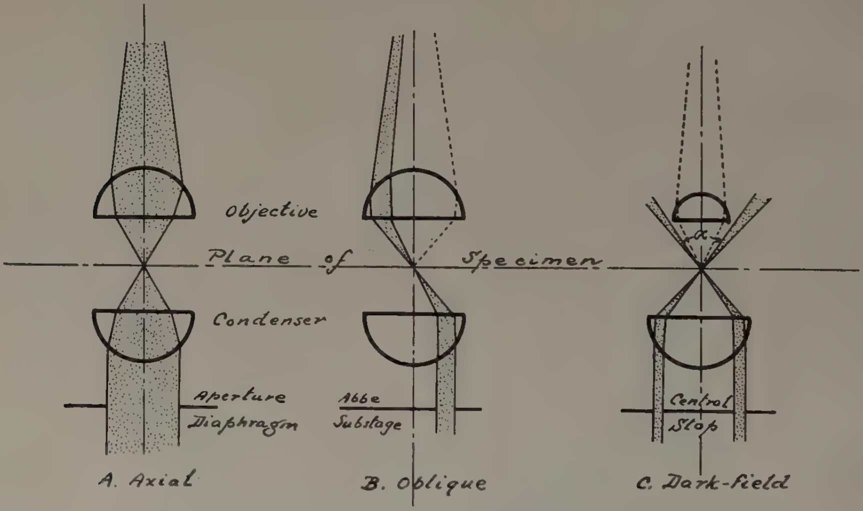

FIG. 23.—COURSES OF LIGHT RAYS IN TRANSMITTED ILLUMINATION.

The circular area at right angles to the optic axis of the microscope, above the eye lens of the eyepiece, where the observer's eye must be placed in order to see the entire field, is called the *Ramsden circle* (Fig. 1). The higher the component magnification of the eyepiece, the smaller the area of the Ramsden circle, and the closer it will be to the eye lens of the eyepiece. This means that it is very difficult to use eyepieces of high magnification, because a slight departure of the eye of the observer from the position of the Ramsden circle will mean obscuration of the field, which induces fatigue.

Further, with high-power eyepieces only a small area of the specimen will be seen, even though highly magnified.

ILLUMINATION

There are three main classes of illumination; incident, transmitted, and vertical.

Incident illumination is that which every-day experience has made familiar. In the microscope using incident illumination, the light is brought to the object from above at some angle to the supporting stage of from 0° to almost 90°. Natural daylight is used as the source of light in most cases, although artificial sources may be employed. Low-power photomicrographs of opaque objects are usually taken with this type of illumination. (See Fig. 4.)

Transmitted illumination is ordinarily employed in observations of transparent preparations such as thin rock sections. Three forms of illumination are included in this class; axial, oblique, and dark-field. Fig. 23 shows the courses of light rays in these three forms of illumination and Fig. 24 shows photomicrographs of a minus 200-mesh galena particle taken with the various types of transmitted light.

Vertical illumination[17] is ordinarily employed in metallography or in the examination of polished opaque mineral surfaces. Three forms of illumination are likewise included in this class; axial,

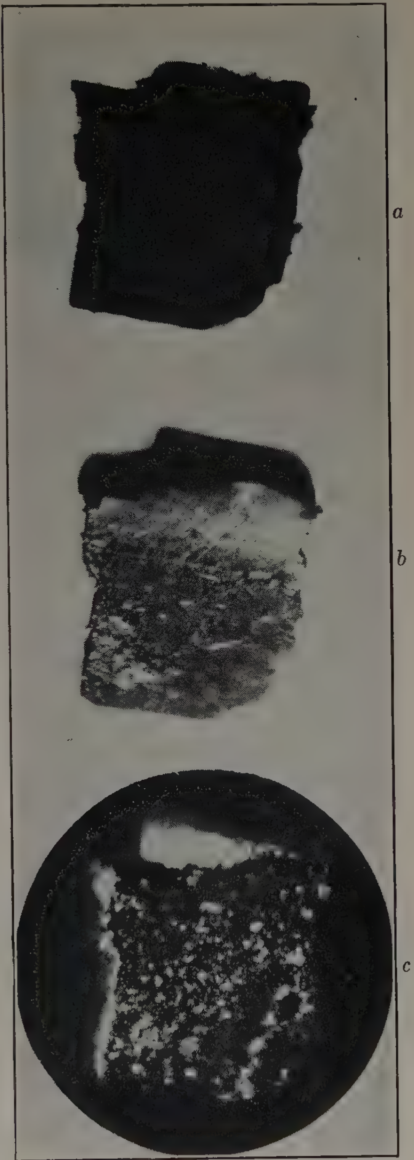

FIG. 24.—MINUS 200-MESH GALENA PARTICLE TAKEN WITH VARIOUS KINDS OF TRANSMITTED LIGHT. × 350.

a. Axial illumination. *b.* Oblique illumination. *c.* Dark-field illumination.

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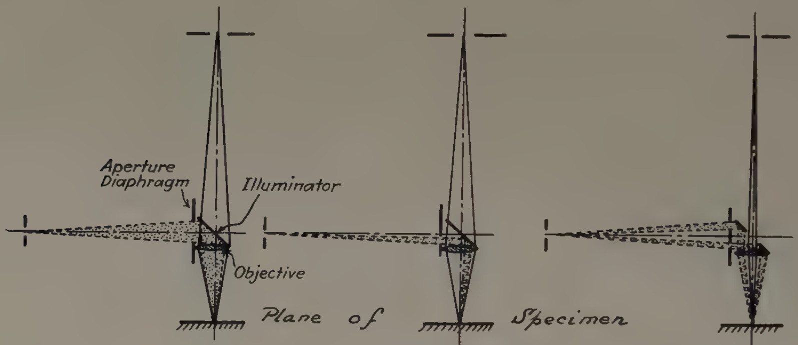
[17] It is interesting to note here that with the vertical illuminator that uses a prism (Nachet), the latter, being opaque to the rays reflected back from the object, cuts the aperture of the objective substantially in half. Therefore, for critical observations, the illuminator that uses the optical flat (Beck) is recommended.

oblique (conical), and dark-field (Ultropak). Fig. 25 shows the courses of the light rays in these three forms of illumination and Fig. 26 shows photomicrographs of slime-coated galena surfaces taken with the various types of vertical illumination. The Ultropak is especially useful in examinations of surfaces, inasmuch as the light from the annular condenser may be focused on different planes parallel to the surface[18] and it further possesses an unusual amount of flexibility (in conjunction with stops) in providing a wide range of sectorial and diaphragmatic illumination. Oblique and dark-field illuminations are very useful also from the point of view of resolving power as it has been predicated above (see page 432).



A. Vertical, Axial B. Vertical, Oblique C. Ultropak

FIG. 25.—COURSES OF LIGHT RAYS IN VERTICAL ILLUMINATION.

In order to obtain good results with any type of illumination, it is necessary to center the illuminating apparatus in order to approximate the conditions pictured in Fig. 3.

CONDENSERS

Since condensers, as stated before, are essentially objectives, they are subject to the same optical defects as the latter. Accordingly, there are condensers possessing different degrees of optical correction. If conditions pictured in Fig. 3 are to be approximated, it must be borne in mind that with an objective with a given degree of optical correction, it is best to use a condenser with a corresponding degree of optical correction.

Although dark-field illumination may be produced as shown in Fig. 23*C* by merely placing a stop below a condenser, the angularity of the emergent beams of light will not be high (angle α of Fig. 23*C* will be relatively small). A good dark field is produced when no direct light enters the objective. Therefore, with the arrangement shown in Fig. 23*C*, owing to the low angularity of the emergent beams, the aperture of the objective cannot be high. In order to permit the use of objectives of relatively high numerical aperture in dark-field illumination with the accompanying increase in resolving power, special dark-field condensers have been designed. These condensers produce emergent beams of high angularity. Hence, objectives of higher aperture may be used without "taking in" any direct light. Objectives having numerical apertures that are too high for the dark-field condensers may be used, provided they are fitted with an iris diaphragm that can be closed so as to give the required reduced aperture.

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[18] See page 400, this volume.

A useful dark-field condenser, which produces emergent beams of such high angularity that objectives with numerical apertures up to 1.05 may be used, is the *Cardioid condenser*. Fig. 27 shows the course of rays in this condenser.

Since the beams in dark-field illumination and in oblique illumination must emerge at angles to the optic axis that are above the critical angle (see page 431 and Fig. 6), the slide must be oiled (with cedarwood oil) to the upper lens of the condenser if the specimen is to receive light.

Technic of Preparation

The most important factor in the successful preparation of a specimen for microscopic observation or for testing in the bubble machine[19] is cleanliness.

Glass slips and cover glasses are cleaned by soaking for several days in cleaning mixture (chromic acid) and then washing in running tap water for several hours. When a slide is prepared, a slip is selected from the container, removed with clean forceps, rinsed in running tap water and in distilled water and then placed on a clean filter paper pad. A drop of the pulp is transferred from the container to the slip with a clean dropper. A few drops of the pulp are allowed to run

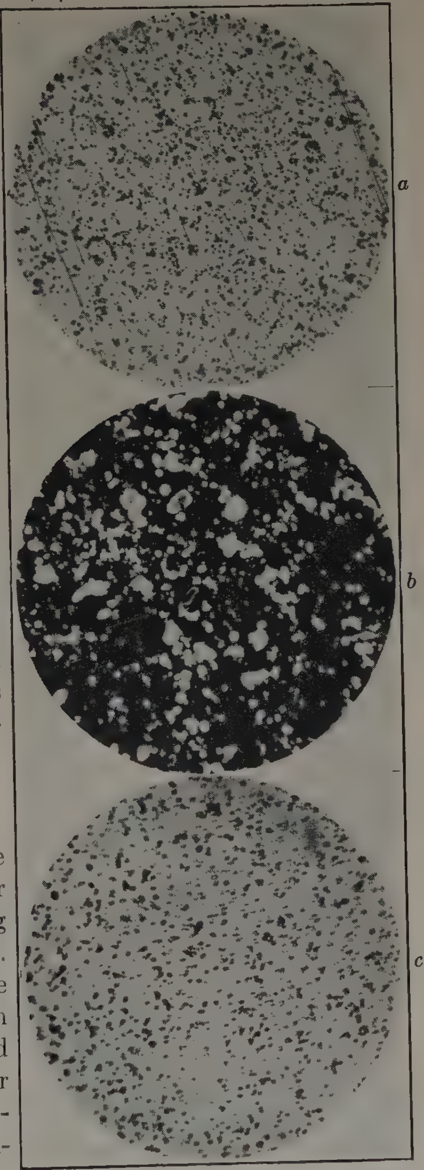

Fig. 26.—Slime-coated galena surface taken with various kinds of vertical illumination.
a. Axial illumination. × 300.
b. Ultropak illumination. × 450.
c. Oblique illumination. × 300.

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[19] Reference of footnote 2.

out before a drop is placed on the slip. The cover glass is also rinsed in running tap water and in distilled water, and then, with clean cover-glass forceps, it is carefully placed on the drop on the slip. In applying the cover glass, one of its edges is first allowed to come in contact with the slip, then the cover glass is allowed to fall upon the drop. When this is carefully done, and the cover glass is clean, the preparation will be free from entrapped air bubbles. The excess liquid is removed with strips of clean filter paper. In order to avoid rapid evaporation of the preparation, the cover glass may be cemented to the slip by sealing with melted paraffin. This is done by applying molten paraffin with a camel's-hair brush, first at opposite corners of the cover glass in order to tack it into place, and then with single, rapid strokes, the preparation is sealed between the cover glass and the slip.[20] A slide so prepared may be kept for days.

Slides of slimes are similarly prepared. They are best examined with oblique or dark-field illumination. For the observation of Brownian movement in slime suspensions,[21] the Cardioid condenser is recommended.

Mineral surfaces or metal surfaces to be tested in the bubble machine or to be observed under the microscope with vertical illumination are prepared in the following manner:

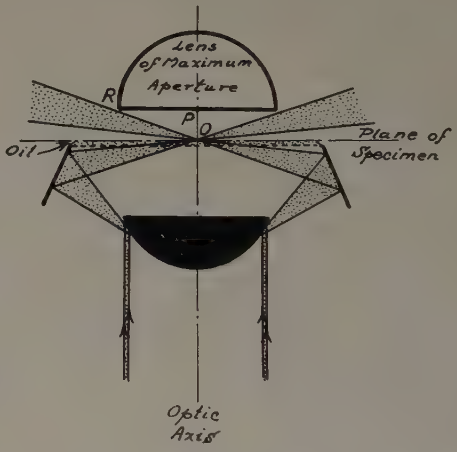

FIG. 27.—COURSE OF RAYS IN CARDIOID CONDENSER.

A piece is selected for its purity, with the rough shape of a cube of ½ in. on a side. Two opposite surfaces are ground flat and parallel on a ground glass, using grade FF alundum and water or similarly suitable grinding media. When reasonably flat and parallel surfaces are obtained, the subsequent polishing is confined to one of the surfaces only. The next step is to polish the surface on a canvas lap with Alundum X (The Norton Co.) and water. The final polish is obtained by using a velvet lap and levigated alumina suspended in running water. This last stage is the most important. The fingers must not come in contact with the surface or the lap (clean, rubber finger tips may be worn) and the surface must be kept constantly under water. When the desired polish has been obtained, the surface is rubbed on a pad of clean filter paper held under water (to remove grinding dust from the polished surface) and finally the particle is placed in a beaker filled with water and in which water is constantly running. The bubble-machine cell, the bubble holder, and the curved dropper must be cleaned with chromic acid and rinsed thoroughly with running tap water and with distilled water. The particles must be handled with platinum-tipped forceps which have been cleaned with chromic acid, rinsed in water and finally burned to a red heat in an oxidizing flame. Under no circumstances should the particle be allowed to dry in air previous to testing in the bubble machine. Clean surfaces in water should give a contact angle of 0° in the bubble machine.[22] If a surface gives a contact angle in clean water, it indicates that it is not clean and it must be repolished (final polish only), until no contact angle is obtained in water. In removing a particle from its container, it must be brought up through a constantly changing air-water interface in order to insure against contamination (air-borne grease will invariably adsorb at stagnant air-water interfaces). Sometimes, when no contact angle is obtained, it may be due to slime particles adhering to the surface.[23] Rubbing on clean filter paper under running water usually removes this slime. It is also of the utmost importance that the water used be reasonably free from heavy metal salts, to avoid possible activation of some mineral particles. Each polished surface prepared should have its own container, in order to avoid activation of some mineral surfaces by salts which may be supplied by other particles.

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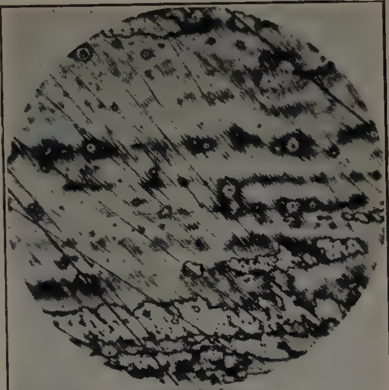
[20] Reference of footnote 8.

[21] Reference of footnote 8.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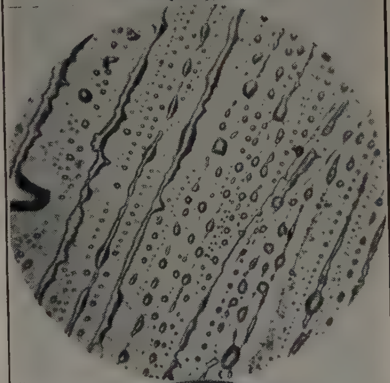
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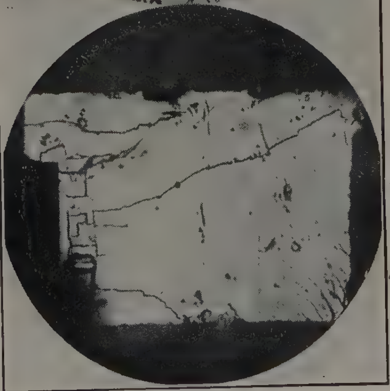
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Fig. 28.—Finger-smeared galena surface. × 300.

Fig. 29.—Finger-smeared quartz surface. × 300.

Fig. 30.—Galena particle from flotation concentrate. Not polished. × 400.

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[22] I. W. Wark and A. B. Cox: Principles of Flotation, see page 196, this volume.

[23] Reference of footnote 4.

Figs. 28 and 29 are polished surfaces of galena and quartz, respectively, which have been finger-smeared with body grease. Note the degree to which grease covers the galena surface while it shows a distinct apathy to spread on the quartz. This seems to indicate that greater care must be used to obtain a "metallic" surface free from grease.

Small mineral particles from flotation concentrates and flotation tailings are usually prepared for microscopic observation by briquetting.[24] Although this method has its merits, and indeed for very fine particles probably is the best method, it has the drawback that particle surfaces as they existed in the pulp cannot be examined as such. A method seldom used, but nevertheless very useful, is to elutriate the flotation products immediately, and then to place a small portion of the solids on a slip. Fig. 30 shows a photomicrograph of a galena particle from a flotation concentrate. The surface has not been polished, therefore it should preserve substantially the same appearance that it had in the original flotation pulp.

The preparation of specimens for slime-coating examination and testing has been thoroughly described in another paper.[25] Again, cleanliness is of the utmost importance.

Examination of mineralized gas bubbles is made by removing a few bubbles from a froth by means of a clean, wide-mouthed dropper and squeezing them in water strongly acidified with sulfuric acid, held in the well of a biological slip. A cover glass is immediately placed on top of the well and cemented thereon with paraffin. The examination is made at relatively low-powers using vertical or incident illumination or a combination of the two. Figs. 9 and 10 are photomicrographs of bubbles so prepared.

ACKNOWLEDGMENTS

Grateful acknowledgment is hereby made to Prof. Arthur F. Taggart for his guidance and constructive criticism. The author is indebted also to E. Leitz, Inc., of New York, for permitting the reproduction of the photograph in Fig. 2.

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[24] R. E. Head and M. Slavin: A New Development in the Preparation of Briquetted Mineral Grains. Univ. of Utah *Tech. Pub.* 10 (1930).

[25] See page 398, this volume.

Soap Flotation of the Nonsulfides*

LIMESTONE, PHOSPHATE ROCK, BAUXITE, FLUORSPAR, RHODOCHROSITE, MANGANESE OXIDES, BARITE, SIDERITE, CHROMITE, SCHEELITE AND FERBERITE, AND CYANITE

BY WILL H. COGHILL,† MEMBER A. I. M. E., AND J. BRUCE CLEMMER‡

(New York Meeting, February, 1932)

FLOTATION has been so closely allied with the sulfide minerals and their early and associated oily reagents that the term "oil flotation" has erroneously been applied to the entire flotation process. Today, the term "chemical flotation" might be used to advantage. Flotation literature has dealt primarily with the removal of sulfides from nonsulfide gangues, and the separation of one sulfide from another. Now, the application of the process to the separation of nonsulfide minerals is coming to the fore; and from the standpoint of the number of commercial minerals involved, the flotation of nonsulfides offers the larger field. Here, soap or soap-forming substances, such as the fatty acids and their derivatives, are used; hence there may well be a subdivision of the chemical flotation process called "soap flotation."

Early experimenters learned that it was easy to form a froth or foam that would carry a part or all of the ore charge over the lip of the flotation machine; but this could not be regarded as flotation because of the lack of selectivity. Doubtless soap was one of the first reagents tried, and doubtless it was rejected on account of its gangue-carrying properties. Now this same gangue-carrying ability makes soap an acceptable reagent, for we are dealing with that which, among sulfides, is considered "gangue." As in the flotation of sulfides, so in the flotation of nonsulfides, the selective flocculation of the mineral and the dispersion of the gangue are the first objectives. In fact, these two phenomena epitomize flotation. They depend upon the unlike surface properties of the respective minerals; and as long as the components of an ore have unlike surface properties, a separation is possible, at least in theory.

Often in nonsulfide flotation it makes little difference which mineral floats and which is depressed; generally only a separation is sought.

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Also, for economic success, the separation does not always have to come up to the standards demanded in sulfide flotation. When two or more minerals in the ore are equally floatable, or when the minerals themselves are not pure, clean concentrates cannot be obtained. These conditions prevail more often in nonsulfide than in sulfide ores. However, some of the nonsulfide ores which have clean mineral grains have yielded soap-flotation concentrates as clean as the best sulfide concentrates. The metallurgical limitations are probably more inherent in the ore than in the soap flotation process.

Flotation research is largely a matter of trial to find the type and amount of reagents. This procedure has aroused a protest from a few scientists who have worried because flotation has not been put into a "scientific straight-jacket"; but it may be said that flotation is as scientific as the world's sciences permit. What science can predict the nature and amount of reagents required to flocculate or disperse ore minerals? Since flocculation of the floatable mineral and dispersion of the gangue are fundamental, the unanswered question suffices to show that the world's sciences, not the flotation engineer, must bear the criticism. If before he entered the laboratory the flotation engineer were to select his reagents and apparatus from a stock of chemicals and an array of intricate equipment, his reports would seem more profound. But he does not so proceed. The flotation machine is at the beginning and end of his experiments. When flotation is achieved the job is finished. This method is sound because flotation cannot be worked out on a test-tube scale. The manner of agitation and beating cannot be duplicated outside of an actual flotation machine, and reagents cannot be split into sufficiently small quantities to permit duplication on very small scale with the accuracy required. For example, in one of the tests to be reported 40 parts of reagent per million parts of water permitted the best work, but 80 parts prevented flotation. Flotation deals with adsorption and solubilities in water in amounts of only a few parts per million. When science has dealt sufficiently with such dilutions, and when, for example, catalyzers are no longer chosen by cut-and-try methods, it will be more consistent to inspect the methods of the flotation engineer. Introspection rather than a desire to create an alibi has prompted these remarks.

Flotation requires an emulsion of air in liquid. The emulsion is made possible and is stabilized by the addition of frothing agents. Pine oil has been the most common. Soap acts in the same capacity, but when used alone it makes a "suds" which is too tough and too stable to be satisfactory. Good flotation requires some bursting and coalescence. The rupture of elastic membranes at the surface often throws the small mineral grains to a distance of several inches. Doubtless similar coalescence of the emulsified air nuclei takes place within the pulp and this aids in dropping entrained gangue. It is fortunate that flotation is best when the bubbles are somewhat brittle, because if they did not break the froth would be so voluminous and permanent that its disposal would be difficult.

ROLE OF SOAP IN FLOTATION

Although, as is commonly known, soap makes a suds, its role in the flotation discussed in this paper seems to be due to its selective adsorption. Selective adsorption implies the use of a bare trace of reagent, and in soap flotation the amount of soap rarely exceeds 100 to 150 parts per million parts of water. This small amount is generally enough to flocculate both the mineral and, to some extent, the gangue. This latter effect must be minimized by using alkaline dispersing agents like sodium silicate or sodium carbonate. Seldom is soap sufficiently selective to be used alone. As will be discussed later, pine oil generally is necessary to impart brittleness to the froth. Consequently, the term "soap flotation" is not strictly technical because the froths are regarded as modified pine-oil froths. (Unless by chance in their experiments the alcohol, used as a solvent for the oleic acid, was a frothing agent, the work reported by Adams[1] and co-authors is strictly soap flotation; and in that way it is unlike most of the work described in this paper.)

If colloid chemists have tested the frothing properties of mixtures of pine oil and soap, they must have reported that in certain ratios these reagents are incompatible. When a dilute soap solution is shaken in a test tube in the presence of an increasing amount of pine oil, its sudsing proclivities gradually disappear until no froth can be formed by shaking. With an excess of pine oil a froth again appears. The flotation tests herein reported were with insufficient pine oil to kill the soap suds, but with enough to enable a good selectivity. The addition of five parts, or more, of pine oil per million parts of water brings about the desired change in selectivity, which is recognized by the bursting of the bubbles before and after the froth has passed over the lip of the machine. The pine oil probably affects the degree of flocculation.

OVERFLOCCULATION

Sometimes the flocculation of the mineral is too intense. In an excellent paper by Ralston and Barker[2] on flocculation, they call this "overflocculation," and in accord with their observations, compact clots of mineral grains have been seen to come to the surface and fall back because they did not retain the quota of air nuclei sufficient to hold them at the surface. Under this condition the gangue also is flocculated, and it contaminates the froth. Phenomena such as these seem to be the only tenets for experimenters who claim that gangue, rather than mineral, should be flocculated. Sometimes the overflocculation may be remedied with alkaline salts. In fact, so much sodium silicate may be used that the entire charge is again dispersed. Sometimes another addition of soap will reinstate flocculation, but it is better to start with a new charge in experimental procedure.

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[1] A. S. Adams, S. M. Kobey and M. J. Sayers: Flotation of Hematite, a Laboratory Success. *Eng. & Min. Jnl.* (1931), **132**, 54.

[2] O. C. Ralston and L. M. Barker: Flocculation and Froth Quality in Flotation. *Preprint* 60-7, Electrochem. Soc. (1931).

Reagents and Machines

Sodium silicate is an important reagent in nonsulfide flotation, but unfortunately the term is not specific. The ratio of soda to silica is seldom considered. Many of the silicates were examined, and, although any number of the series may be used, the ortho-silicates and meta-silicates were preferred. Bicarbonates are undesirable as alkaline dispersants. In fact, suspicion has rested on the carbon dioxide naturally contained in the air passed into the pulp. To determine the effect of carbon dioxide comparative flotation runs were made with natural and with "scrubbed" air. The latter, where the carbon dioxide had been removed by passing the air through a solution of caustic, gave the better results. If this observation is correct, pneumatic machines that employ contaminated air as a means of agitation should be avoided in soap flotation.

Fatty acid reagents are changed instantly upon entering hard water. Very few nonsulfide ores are without the accessory soluble salts that make water hard. Some hardness is allowable but the excessive amount of hardness induced by nonsulfide ores from the arid Southwest has hindered good flotation. In the study of the effect of hard water, calcium oleate was prepared and used as a flotation agent. The flotation was satisfactory. An elevated temperature seems to make hard water more tolerable.

The nonsulfide minerals which possess distinct crystalline structure and luster are the most floatable. Earthy minerals are generally difficult to concentrate. Wad manganese ores are especially difficult.

As would be expected, hydrogen ion determinations are difficult in pulps with the gangue so thoroughly dispersed as in soap flotation.

Strangely, sulfuric acid has been a valuable reagent in some of the soap flotation of nonsulfides. Sometimes it is used in the cleaning of the rougher concentrate; but when the ore is free from gangue slime, it may be used advantageously in the rougher. When coarse gangue contaminates the cleaner concentrate a trace of dilute sulfuric acid will depress it.

Mechanical machines with a positive means for controlling the air are best. In some of the mechanical machines the leak of air through the stuffing box which constitutes a part of the aeration unit is objectionable because it prevents regulation of air through the valve. Mechanical beating in the presence of a moderate feed of air is best in soap flotation.

In the work reported in the following pages vertical-shaft mechanical agitation flotation machines were used. Air machines gave too much suds for soap flotation. The pulp densities were in accord with usual practice.

LIMESTONE

As early as January, 1925, a flotation test on calcareous iron ore showed that the limestone was floatable with a fatty acid. Subsequently Lee[3] used oleic acid and cresol in the flotation of limestone from magnetic log-washer tailing. The iron ore, which was the source of the tailing, had previously received a reducing roast at 550° C., and the iron had been removed magnetically. The tailing from the magnetic machines was high in limestone and silica. Since the limestone was needed as a flux in the blast furnace it was desirable to recover it by flotation and add it to the iron concentrate. The siliceous flotation tailing was sent to waste. By this means a self-fluxing blast-furnace feed was prepared. A point of technical interest is that flotation worked satisfactorily on a feed that had been subjected to an elevated temperature, although the temperature was not high enough to calcine the limestone. The absence of sulfides probably favored the process.

PHOSPHATE ROCK

During the course of an investigation of the methods of washing the phosphate rock of the land-pebble district of Florida, soap flotation was tried on the fines in the waste. The results are reported by Lawrence and DeVaney.[4] The phosphate rock was concentrated and a fine quartz sand tailing was rejected. Soap, pine oil, sodium silicate and sodium carbonate were the chief reagents. Under the protection of patents one of the Florida companies has been practicing, intermittently at least, soap flotation of phosphate rock.

In the further pursuance of this investigation Lawrence and Roca[5] wrote a paper. They continued the use of soap but replaced pine oil with sodium sulfide.

The notes on the flotation of phosphate indicate that the flocculation of the phosphate must have been intense, while the sand grains were dispersed. Lawrence and Roca make the following statement:

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[3] O. Lee: Flotation of Limestone from Siliceous Gangue; U. S. Bur. Mines *Rept. of Investigation* 2744 (1926).

[4] H. M. Lawrence and F. D. DeVaney: Flotation of Low-grade Phosphate Ores. U. S. Bur. Mines *Rept. of Investigation* 2860 (1928).

[5] H. M. Lawrence and E. Roca: Flotation of Low-grade Phosphate Ores, II. U. S. Bur. Mines *Rept. of Investigation* 3105 (1931).

> As an alternative to the grinding and flotation of the coarser grains, the possible application of a panning action for the recovery of these coarser grains was studied. In experiments performed during the winter of 1927–28 it had been observed that grains of phosphate could be separated from the sand grains by panning after treatment with flotation reagents. Tailings from flotation tests of unground sands were panned, and concentrates analyzing 66 to 68 per cent bone phosphate frequently were obtained. In this panning action the phosphate grains passed over the lip of the pan partly as individual grains or as a scum of flocculated grains and partly by rolling over the grains of sand that settled in the pan.

This quotation is in accord with the views about flocculation and dispersion as set forth in this paper, and the hint about gravity concentration is pertinent. Table concentration of phosphate rock after conditioning in reagents commonly classed as flotation reagents has received a commercial trial in Florida, but the authors are not informed as to the outcome of the test. In the new process the intention is to bring the phosphate grains into large floccules, so that the sizing action of the table will remove them at the tailing side and carry the dispersed sand grains to the concentrate end. With quartz and phosphate the specific gravity relation is more favorable than it would be were the flocculated mineral heavy.

BAUXITE

Bauxite ores are among the most unpromising for flotation. The report by Gandrud and DeVaney[6] shows that they may contain clay, hematite, limonite, siderite and quartz. Some of these gangue minerals are floatable. Hence the flotation of bauxite is difficult.

Sodium sulfide and oleic acid were the reagents. The froths were more voluminous than if pine oil had been used. No theory has been forthcoming to justify the use of sodium sulfide, and it has not been employed in any of the tests that follow.

FLUORSPAR

The flotation of fluorspar long since has passed the experimental stage. Soap and pine oil, or the equivalent of the latter, are fundamental reagents. In the paper by Coghill and Greeman[7] the results of preliminary experimentation are given, as is also a warning against hard water because of its action on soap. Plant operations, however, have proved this statement to be overly cautious, because moderately hard water has since been used successfully. This is natural, because calcium and magnesium oleates, the reaction products of soap with hard water, are known to be moderately soluble.

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[6] B. W. Gandrud and F. D. DeVaney: Preliminary Examination of Low-grade Bauxite with Particular Reference to Flotation. U. S. Bur. Mines *Rept. of Investigation* 2906 (1928).

[7] W. H. Coghill and O. W. Greeman: Flotation of Fluorspar Ores for Acid Spar. U. S. Bur. Mines *Rept. of Investigation* 2877 (1928).

RHODOCHROSITE

Before the application of flotation, veins of rhodochrosite containing quartz and other gangue minerals were regarded as waste rock. Now they are valuable orebodies. The flotation concentrate is of good grade and it can be enriched further by calcining to drive off the carbon dioxide. When the ore contains sulfides, they can be floated ahead of the manganese mineral to produce a sulfide concentrate. The laboratory results are reported by DeVaney and Coghill.[8] Soap and pine oil were the basic reagents. The laboratory results have been confirmed by commercial runs. When the rhodochrosite veins contain other carbonates the success of flotation will be impaired because all carbonates are floatable.

MANGANESE OXIDES

Some of the manganese oxides are so permeated with gangue that liberation by grinding is impossible. Some of them in the arid districts, as in the Southwest, contain excessive amounts of soluble salts such as gypsum. When the soluble salts have had time to dissolve in the pulp they may cause trouble. On the other hand, some of the manganese oxides are amenable to flotation. Notice of this was given by DeVaney and Clemmer.[9] Their forecast has been substantiated by the commercial flotation unit in the Cuyuna district, Crosby, Minnesota.

BARITE

Barite generally occurs in residual deposits associated with clay, quartz, and iron oxides. The clay can be removed by log washing but the iron oxides and quartz require some other method. Barite is one of the most floatable of nonsulfides, but oleic acid alone will not give good concentrates. A small amount of alkali, either sodium carbonate, hydroxide or silicate readily disperses the gangue and induces a richer froth. Too much dispersing agent gives an undesirable "watery" froth. Losses of slimed mineral increase with the alkali, and complete dispersion may result should the amount of alkali be excessive. Sodium silicate requires closer reagent control than the carbonate or the hydroxide.

The results of a test on an Arkansas ore are shown in Table 1. The reagent list shows that sodium silicate was used in the cleaning. The iron, which is often penalized by the buyer, was reduced from 0.65 to 0.11 per cent. Not all barite ores are amenable to flotation. Other sulfates or carbonates float too freely and they also will go into the concentrates and reduce the grade.

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[8] F. D. DeVaney and Will H. Coghill: Preliminary Ore-dressing Tests to Recover Manganese in Rhodochrosite Ores. U. S. Bur. Mines *Rept. of Investigation* 2902 (1928).

[9] F. D. DeVaney and J. B. Clemmer: Floating of Carbonate and Oxide Manganese Ores. *Eng. & Min. Jnl.* (1929) **128,** 506–508.

TABLE 1.—*Arkansas Barite Ore*

Product	Weight, Per Cent	Assay, Per Cent			Per Cent, Total		
		$BaSO_4$	SiO_2	Fe	$BaSO_4$	SiO_2	Fe
Cleaner concentrate	70.5	97.25	1.76	0.11	77.8	16.2	11.9
Cleaner tailing	18.4	83.95	10.87	0.63	17.5	26.1	17.9
Rougher concentrate	88.9	94.49	3.64	0.22	95.3	42.3	29.8
Rougher tailing	11.1	37.40	39.90	4.08	4.7	57.7	70.2
Composite (feed)	100.0	88.16	7.67	0.65	100.0	100.0	100.0

Reagents Used	Pounds per Ton of Crude Ore	
	Rougher	Cleaner
Pine oil	0.08	
Oleic acid	0.32	
Sodium carbonate	1.00	
Sodium silicate (feed)	0.50	1.00

SIDERITE

On account of the interest of investors in a body of siderite in Texas, a sample was sent to the Bureau of Mines for examination. Although siderite, like other carbonates, is readily floatable, the sample gave trouble because the gangue contained a partly water-soluble mineral resembling glauconite. Quantitative tests showed that some lime and a large amount of magnesia dissolved in distilled water. The ore required grinding to 48 mesh for liberation. Removing the water contaminated during wet grinding and using fresh water in flotation gave good results. Precipitating from the contaminated water the soluble salts, by a lime-soda ash or a caustic soda treatment, also allowed good results to be obtained in flotation. As the precipitate in the latter method did not cause trouble, it was not removed from the pulp.

Table 2 shows the results of a flotation test on −48-mesh siderite. The object of the hydroxide added in the rougher was to precipitate the magnesia. The hydroxide and silicate used in the cleaner were to depress the slimy gangue. The high iron content of the tailing is not evidence against the flotation, because glauconite, which constituted the tailing, is known to contain about 25 per cent of iron in chemical combination. Flotation of a feed as coarse as 48 mesh requires care in regulating the amount of alkali, as a slight excess tends to depress the +65-mesh grains. Calcination of the concentrate graded it up to 63.75 per cent of iron and 6.65 per cent of insoluble.

TABLE 2.—*Texas Siderite Ore*

Product	Weight, Per Cent	Assay, Per Cent		Per Cent, Total	
		Iron	Insol.	Iron	Insol.
Cleaner concentrate	48.0	44.44	5.20	59.4	13.5
Cleaner tailing	14.1	33.13	23.80	13.0	18.1
Rougher concentrate	62.1	41.87	9.42	72.4	31.6
Rougher tailing	37.9	26.18	33.40	27.6	68.4
Composite (feed)	100.0	35.93	18.51	100.0	100.0

Reagents Used	Pounds per Ton Crude Ore	
	Rougher	Cleaner
Pine oil	0.08	
Oleic acid	0.32	
Sodium oleate	0.40	
Sodium hydroxide	1.00	0.75
Sodium silicate		0.50

CHROMITE

The only important chromium mineral is chromite. It is seldom pure and may contain from 40 to 68 per cent of chromic oxide. Ore samples from California and Montana were tested.

California Chromite

A sample of California chromite contained small crystals of the mineral disseminated in gangue predominantly serpentine (hydrous magnesium silicate). The clean mineral grains contained 55 per cent of chromic oxide. Several preliminary flotation tests fell short of expectations; concentrates of good grade were made with little difficulty but the recovery was low. The reagent charge included nominal amounts of pine oil, oleic acid, sodium oleate and sodium carbonate. Soon after starting, the froth became so unstable that no more mineral floated. Addition of more reagent failed to assist in promoting flotation.

Further tests showed that the serpentine or some other gangue mineral was somewhat soluble in water; hydrogen ion tests on water after contact with the ore revealed that the water was decidedly alkaline. Qualitative tests showed some calcium and a large amount of magnesium carbonate or bicarbonate salts in solution. Similar tests conducted in the presence of sodium carbonate revealed that it accelerated the rate of solution of the gangue. Thereafter carbonates were omitted and an extra amount of soap and sodium hydroxide was used. It seems that magnesium hydrate precipitated in the pulp does not interfere, whereas magnesium carbonate is injurious.

The ore was ground to 100 mesh in the presence of some oleic acid and sodium oleate. A little sodium hydroxide assisted in the cleaning treating. In some of the tests chromite as coarse as 48 mesh was floated. The change from former procedure proved beneficial, as shown by the results in Table 3.

TABLE 3.—*California Chromite Ore*

Product	Weight, Per Cent	Assay, Per Cent Cr_2O_3	Per Cent Total Cr_2O_3
Cleaner concentrate	39.5	52.95	89.5
Cleaner tailing	11.8	11.89	6.0
Rougher concentrate	51.3	41.22	95.5
Rougher tailing	48.7	2.17	4.5
Composite (feed)	100.0	23.37	100.0

Reagents Used	Pounds per Ton Crude Ore		
	Grind	Rougher	Cleaner
Pine oil		0.08	
Oleic acid	0.60	0.15	
Sodium oleate	0.75	0.25	
Sodium hydroxide			0.75

It is of interest to note that acid may be used in the soap flotation of chromite. Recent tests made on a sample of California beach sands gave good results when a small amount of sulfuric acid was used after conditioning with soap. The feed was a high-intensity magnetic separator product enriched in chromite and garnet. The absence of gangue slime made possible the use of acid in both roughing and cleaning operations and flotation yielded chromite concentrates of good grade and recovery. The garnet was depressed more readily with acid than with sodium carbonate or silicate. This phase of soap flotation warrants further investigation.

Montana Chromite

The mineral in the Montana chromite is present as small crystals disseminated in a gangue of compact serpentine with minor amounts of calcite and dolomite. The chromite is lower in grade than that from California. Selected grains contained 45 per cent of chromic oxide. The difficulties experienced with the first California sample, on account of soluble salts, were less prominent with this ore; the compact serpentine gangue exhibited a tendency to go into solution, but to a lesser degree. However, the exchange of bases was evident in flotation tests with sodium carbonate as the alkali. After a short time the pulp became unstable and flotation ceased. Caustic soda, when used in closely regulated amounts, allowed the flotation to proceed in regular fashion for a sufficient time to give the desired recovery. Results of a typical test are given in Table 4. The grade of the concentrate is somewhat vitiated by mineral carbonates in the ore.

TABLE 4.—*Montana Chromite Ore*

Product	Weight, Per Cent	Assay, Per Cent Cr_2O_3	Per Cent Total Cr_2O_3
Cleaner concentrate	76.3	43.69	92.2
Cleaner middling	10.9	20.74	6.2
Rougher concentrate	87.2	40.82	98.4
Rougher tailing	12.8	4.47	1.6
Composite (feed)	100.0	36.17	100.0

Reagents Used	Pounds per Ton Crude Ore	
	Rougher	Cleaner
Pine oil	0.08	
Oleic acid	0.32	
Sodium oleate	0.75	
Sodium hydroxide	1.50	0.50
Sodium silicate		1.00

SCHEELITE AND FERBERITE

Two types of tungsten ores were examined, scheelite ($CaWO_4$) from Nevada, and ferberite ($FeWO_4$) from Colorado.

Nevada Scheelite

The Nevada scheelite ore was hard and had a quartz gangue. It was ground to 100 mesh. The results of flotation are shown in Table 5. In another test the concentrate was graded up to 79 per cent tungstic oxide (scheelite contains 80.6 per cent of tungstic oxide) but the recovery was a little lower than that shown in the table. Another sample with some sulfides yielded first a sulfide concentrate and then a tungsten concentrate. The use of the sulfide flotation reagents did not interfere with the subsequent soap flotation.

TABLE 5.—*Nevada Scheelite Ore*

Product	Weight, Per Cent	Assay, Per Cent WO_3	Per Cent Total WO_3
Cleaner concentrate	3.4	62.72	89.7
Cleaner tailing	21.7	1.03	9.4
Rougher concentrate	25.1	9.38	99.1
Rougher tailing	74.9	0.03	0.9
Composite (feed)	100.0	2.38	100.0

Reagents Used	Pounds per Ton Crude Ore	
	Rougher	Cleaner
Pine oil	0.12	
Oleic acid	0.16	
Sodium oleate	0.40	
Sodium carbonate	0.75	0.75
Sodium silicate		0.40

In judging scheelite froths, the kind of illumination is important. Scheelite is florescent in ultra-violet rays. The florescence induced by an ultra-violet light made it easily distinguishable from quartz.

Colorado Ferberite

Two samples of Colorado ferberite were examined, a massive ore and a mill slime. In the first sample the mineral was so finely disseminated in quartz gangue that crushing to 200 mesh was required. The results of flotation are given in Table 6. The rougher concentrate was cleaned three times and the cleaner tailing is shown as "composite cleaner tailing." Whereas alkali is required for good roughing flotation, an excess destroys flotation. In the cleaning the usual practice was departed from by using sulfuric acid. This anomalous practice can be justified only by the results. Sulfuric acid dispersed the remaining gangue and gave a concentrate of better grade. A slight excess destroyed flotation; the right amount gave a pulp just slightly acid. When acid is used in the cleaning some additional pine oil may be required. The technique of repeated cleaning in the laboratory is difficult because the charge is so small. A larger amount of crude ore would have to be used for better results.

TABLE 6.—*Colorado Ferberite Ore*

Product	Weight, Per Cent	Assay, Per Cent WO_3	Per Cent Total WO_3
Cleaner concentrate	13.7	56.00	77.4
Composite cleaner tailing	33.6	5.59	18.9
Rougher concentrate	47.3	20.28	96.3
Rougher tailing	52.7	0.69	3.7
Composite (feed)	100.0	9.91	100.0

Reagents Used	Pounds per Ton Crude Ore			
	Rougher	Cleaner		
		No. 1	No. 2	No. 3
Pine oil	0.08			0.04
Oleic acid	0.24			
Sodium oleate	0.60			
Sodium hydroxide	0.75			
Sodium silicate		0.20		
Sulfuric acid			0.16	0.16

The second sample was a mill slime, the screen analysis of which is shown in Table 7. The mineral was excessively fine and the sample was weathered. The gangue was quartz and some altered feldspars. The flotation results are given in Table 8, and show a very small loss in the tailing, but the recovery was not good; the four cleanings left too much mineral in the middling. Doubtless plant operation would improve the technique enough to add to the recovery. The phosphorus content of the concentrate was not determined.

TABLE 7.—*Screen Analysis of Colorado Ferberite Mill Slime*

Size	Weight, Per Cent	Assay, Per Cent WO_3	Per Cent Total WO_3
Mesh			
+100	3.2	1.33	0.5
100 to 200	15.1	1.61	3.5
200 to 400	25.1	2.06	7.5
−400	56.6	21.55	88.5
Composite (feed)	100.0	13.00	100.0

TABLE 8.—*Colorado Ferberite Mill Slime*

Product	Weight, Per Cent	Assay, Per Cent WO_3	Per Cent Total WO_3
Cleaner concentrate	11.8	49.90	46.2
Composite cleaner tailing	45.3	14.31	50.9
Rougher concentrate	57.1	21.66	97.1
Rougher tailing	42.9	0.87	2.9
Composite (feed)	100.0	12.74	100.0

Reagents Used	Pounds per Ton Crude Ore				
	Rougher	Cleaner			
		No. 1	No. 2	No. 3	No. 4
Pine oil	0.08				0.04
Oleic acid	0.24				
Sodium oleate	0.60				
Sodium hydroxide	0.30	0.20			
Sodium silicate		0.20			
Sulfuric acid			0.08	0.16	0.16

CYANITE

A good concentrate of cyanite ($Al_2O_3 \cdot SiO_2$, 63 per cent Al_2O_3, sp. gr 3.61) was made from an ore with clean quartz gangue. The sample was crushed to 100 mesh. The results in terms of Al_2O_3 are shown in Table 9. The specific gravities of the concentrate and tailing are also given. Another sample with quartz, biotite, muscovite and undetermined varieties of garnet gave more trouble. Some sulfuric acid had to be used in the cleaning to depress the biotite and garnet. The quartz and muscovite were readily eliminated in the rougher. The results with this second ore are shown in Table 10. On account of the variety of aluminous minerals the grade could not be determined by chemical analysis; specific gravity determinations were used.

OTHER MINERALS

Other of the silicate minerals, such as spodumene and beryl, have been found amenable to concentration by soap flotation methods. The gangues were composed principally of quartz. Although the work is of a preliminary nature, the results are gratifying in that concentrates of fair grade can be produced. The recovery of mineral is not great; and the slight difference in floatability existing between these silicate minerals

TABLE 9.—*Cyanite Ore No. 1*

Product	Specific Gravity	Weight, Per Cent	Assay, Per Cent Al_2O_3	Per Cent Total Al_2O_3
Cleaner concentrate	3.58	15.4	61.20	88.7
Cleaner tailing		1.5	34.30	4.8
Rougher concentrate		16.9	58.82	93.5
Rougher tailing	2.66	83.1	0.82	6.5
Composite (feed)		100.0	10.62	100.0

Reagents Used	Pounds per Ton Crude Ore	
	Rougher	Cleaner
Pine oil	0.16	
Oleic acid	0.32	
Sodium oleate	0.60	
Sodium hydroxide	1.00	0.50
Sodium silicate		0.50

TABLE 10.—*Cyanite Ore No. 2*

Product	Weight, Per Cent	Specific Gravity
Flotation concentrate	22.8	3.59
Flotation middling	14.2	3.17
Flotation tailing	63.0	2.97
Composite (calculated)	100.0	3.14
Feed		3.13

Reagent Used	Pounds per Ton Crude Ore		
	Rougher	Cleaner	Recleaner
Pine oil	0.08		
Oleic acid	0.32		
Sodium oleate	0.60		
Sodium hydroxide	1.00	0.75	
Sodium silicate		0.75	
Sulfuric acid			0.32

and quartz demands a precise control of reagents if a separation is to be obtained, but tests have demonstrated the possibility of such a separation.

CONCLUSIONS

Intermittently the study of soap flotation of nonsulfide ores has been in progress in the Bureau of Mines since 1925. In terms of number of ores, nonsulfide flotation is more important than sulfide flotation. Though incomplete, the investigation has included fourteen "ores": limestone, phosphate rock, bauxite, fluorspar, rhodochrosite, manganese oxides, barite, siderite, chromite, scheelite, ferberite, cyanite, beryl and spodumene. Of these, four have been put through successful trials in commercial flotation plants; *i. e.*, phosphate rock, fluorspar, rhodochrosite and manganese oxides. Laboratory tests indicate that some of the other ores mentioned are equally amenable.

The work has been done under a cooperative agreement between the Mississippi Valley Experiment Station of the United States Bureau of Mines and the Missouri School of Mines and Metallurgy, Rolla, Mo., and between the Southern Experiment Station of the United States Bureau of Mines and the University of Alabama, Tuscaloosa, Alabama.

The flotation of such ores depends on the proper selection and amounts of pine oil, sodium oleate, oleic acid, sodium carbonate, sodium hydrate, sodium silicate and similar reagents. That sulfuric acid has to be added to this list of alkalies seems an anomaly, but repeated tests have shown that some of the ores require a little sulfuric acid in the cleaning treatment. Generally less than one hundred parts of any one of the reagents in one million parts of water is sufficient. A slight excess is likely to destroy flotation.

Often the worker may be guided by phenomena instantly observable to the naked eye; and observations throughout the work lend justification to the belief that the principles of flotation are epitomized in the "flocculation of the mineral and dispersion of the gangue."

DISCUSSION

(*Arthur F. Taggart presiding*)

A. M. GAUDIN,* Butte, Mont. (written discussion).—The authors are to be congratulated on a timely presentation, in the form of a summary, of a subject of growing industrial possibilities. There is little doubt that soap flotation of the nonsulfides will develop in the future to substantial magnitude, thanks to the efforts of pioneers in this field.

There is little ground for disagreement with the authors on their facts, as much of the work—e.g., that on cyanite—has been done in an entirely new field of endeavor. On the other hand, possibly in order to shorten their paper, the authors do not appear to be interested in giving a scientific interpretation to their otherwise most fascinating facts. They would have others believe that such a graduation from rule-of-thumb methods is tantamount to becoming imprisoned in a straight-jacket.

Perhaps it is too early in the development of the art to attempt a complete interpretation of the facts, and yet it would seem that there is enough evidence available to show that soap flotation of nonsulfides results from the formation at the mineral surface of insoluble soaps by reaction of metal ions at the mineral surface with fatty acid ions in the solution. It is also incontrovertible that close pH control is prerequisite for successful selection. Yet pH observations are not recorded by the authors.

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The evidence as to the action of soap on nonsulfide minerals is largely to be had from the experiments quoted by the authors, but also from direct determinations of abstraction of soap by apatite,[10] and by many thousands of experiments on the actual flotation behavior of simple nonsulfide systems.[11] Quite improperly, it would seem, the authors would exclude from the field of flotation those experiments dealing with the flotation of pure minerals. Yet these experiments represent a limiting case of no direct, but of immense indirect, practical value.

P. Malozemoff and W. D. Wilkinson, Butte, Mont. (written discussion).—Various investigators,[12] as well as the authors of the paper under discussion, have made generalized statements concerning the major dependence of flotation on flocculation and dispersion. Because of inadequate evidence and indefinite experimental methods, these statements are not entirely convincing.

There are three provinces in which the study of flocculation in flotation is possible; namely, the pulp, the froth, and the disintegrated froth. In each case, the conditions and forces that act on the mineral vary, because the proportional relations of the several phases (air, liquid and solid) as well as of the addition agents are not the same. For instance, it is possible that a mineral be flocculated in the pulp, in the disintegrated froth, but not in the froth before the froth is disintegrated.[13] It is also possible that other combinations may occur.

As a criterion of flotation behavior, observations in any one of the three provinces are incomplete, and statements as to the necessity of flocculation for successful flotation must take into account all three. Furthermore, it is advisable to distinguish the use of the word "flocculation" as meaning the "coalescence of mineral particles around gas nuclei," from the accepted physicochemical use as a "mutual adherence of the mineral particles."

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[10] N. Luyken and E. Bierbrauer: The Recovery of Apatite from Waste Ore Slimes by Flotation. *Mitt.* Kaiser Wilhelm Inst. Eisenforsch. Duesseldorf, **10,** 317–321.

[11] A. M. Gaudin, H. Glover, M. S. Hansen and C. W. Orr: Flotation Fundamentals, I. Utah Eng. Expt. Sta. *Tech. Pub.* 1 (1928).

[12] H. L. Sulman: A Contribution to the Study of Flotation. *Trans.* Inst. Min. & Met. (1919–1920) **29,** 44; O. C. Ralston and S. M. Barker: Flocculation and Froth Quality in Flotation. Electrochem. Soc. *Pre.* 60–7.

[13] The bubble walls in certain froths have been definitely observed to be monoparticular and, therefore, not flocculated.

Milling Methods and Costs at the No. 2 Concentrator of the Phosphate Recovery Corporation

By H. S. Martin,* Mulberry, Florida

(New York Meeting, February, 1933)

The Phosphate Recovery Corporation operates three flotation plants, Nos. 1 and 2 concentrators about three miles northeast of Mulberry, Florida, and No. 3 plant at Wales, Tennessee. These plants represent the first commercial phosphate flotation operation and are also believed to be the largest nonmetallic flotation operation in the world at the present time.

The details of this paper will be confined to plant No. 2, this being the newest and largest concentrator in Florida, and the flow sheet being practically the same as at plant No. 1. The No. 3 plant has been operated but a short time and has been shut down since December, 1932, on account of the decrease in phosphate rock production.

The phosphate washers usually are located centrally on a known deposit so that about two hundred acres of ground can be mined economically to each washer location. The washers are moved to new areas when the distance becomes too great for economical pumping. The flotation plants are built either adjacent to a washer, so that current debris may be utilized, or on the site of a former washer where there is sufficient accumulated debris available for mining, and the flotation plants must also be moved from time to time.

Most of the water used in the Florida flotation plants is recovered after passing through a circulating system where most of the fine suspended matter is settled out, but some make-up water is obtained from deep wells near the plant.

Power is obtained from the Tampa Electric Co. through the distribution facilities of the International Agricultural Corporation.

Flotation concentrate is hauled from the plant by trolley over the International Agricultural Corporation's trolley system, to the drying plant at Prairie, one mile northwest of Mulberry.

Ore Treated

In the pebble phosphate district of Florida, the phosphate mineral, or collophane, is usually found in horizontal beds of varying thickness covered by varying depths of overburden. At present the overburden is usually removed by electric dragline and the actual mining of the phosphate matrix is done by hydraulic means. The phosphate pebbles and grains usually are embedded in a matrix of sand and clay and the methods of washing and screening have been described frequently.

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* Manager, Phosphate Recovery Corporation.

The older washing methods are briefly described as follows: Hydraulic guns using water at 200 lb. per square inch cut into and disintegrate the "matrix," which is then carried by the water to a pump sump, whence it is pumped by 10-in. or 12-in. sand pumps to the washer. The pump discharge passes first through a trommel screen, and all plus 1½-in. material, mud balls and rock, are rejected. Undersize from the trommel usually passes over a flat screen of slotted metal, with openings $\frac{3}{32}$ to $\frac{3}{64}$ in. wide. Undersize from the flat screen is washer tailing or "debris." The partly dewatered oversize from the flat screen usually passes over vibrating screens and thence through a series of two spirally bladed "log-washers" that break up remaining lumps of stiff clay. Overflow from both log-washers is also waste. The rake discharge from the second log-washer passes over vibrating screens, where it is separated into two sizes of finished product, fine and coarse pebble. A more complete description of this part of the process was given in *Pit and Quarry* for Dec. 30, 1931.

After the clay is worked into suspension the washer process is one of size separation, most of the phosphate grains being larger than the quartz grains with which they are associated. In every deposit, however, there is more or less phosphate rock of approximately the same size as many of the sand grains, and size separation for this material is impossible. The specific gravity of phosphate particles and quartz particles is so nearly the same that gravity concentration methods are not feasible. All such material was formerly wasted by the washers, and was called "debris." It is this "debris" that furnishes feed for the flotation plants.

The amount of rock formerly wasted varies in different localities, but in a great many instances represents a tonnage equal to the tonnage of "pebble" originally obtained by the washer. Concentration of this finer material therefore will almost double the tonnage of output from a given area of matrix. It is also true that the "coarse pebble" usually contains inclusions of quartz, clay and feldspar and is, consequently, of lower grade than the flotation concentrate made from the finer portions of the same matrix, particularly if the flotation feed is ground.

Feed for the No. 2 plant is obtained by remining several old debris ponds. Most of the debris consists of quartz and phosphate grains ranging in size from minus 8 mesh to plus 200 mesh, although there is some material larger than 8 mesh and some smaller than 200 mesh. At the time the debris was discharged by the old washers a great deal of clay was carried away from the sandy material by the water and deposited at a distance farther away from the washer site. Hydraulic mining is used in handling this debris, although the water pressure here is only 50 to 60 lb. per square inch. At the No. 1 plant current washer debris from the No. 11 washer of the International Agricultural Corporation is used as feed for flotation and is pumped as produced to the flotation feed bins.

Although at flotation size (minus 28 to minus 35 mesh) there are very few apparent inclusions of quartz in the phosphate particles, the chemical composition of the rock seems to vary in different localities. Apparently clean phosphate particles from one deposit may assay as low as 68 to 70 per cent $Ca_3(PO_4)_2$ (bone phosphate of lime) while similar grains from another locality may assay as high as 78 to 80 per cent bone phosphate of lime. Collophane, which is the predominant mineral in these deposits, is generally considered to be a complex calcium carbono-fluo-phosphate, the fluorine and carbon dioxide being tied up molecularly with the phosphate. Separation of these constituents is impossible, of course, by flotation, the main object in flotation being to separate the collophane particles from the quartz particles.

TABLE. 1.—*Screening Analysis of Crude Debris as It Comes from Pit*

PHOSPHATE RECOVERY CORPORATION, PLANT NO. 2

Mesh	Weight, Per Cent	Weight, Cum. Per Cent	Bone Phosphate of Lime, Per Cent
Original	100.00		24.79
+ 10	2.58	2.58	55.44
+ 14	1.41	3.99	73.62
+ 20	3.75	7.74	73.52
+ 28	8.43	16.17	54.64
+ 35	19.20	35.37	27.02
+ 48	23.65	59.02	16.77
+ 65	21.31	80.33	12.76
+100	10.77	91.10	13.09
+150	4.45	95.55	9.44
−150	1.17	96.72	26.52
Slime ([a])	3.28	100.00	28.42

[a] Sample stirred in pail with water at 15 to 20 per cent solids, allowed to settle 10 seconds, then decanted. Operation repeated three times.

In a rather complete investigation made in 1929 by Prof. R. J. Colony and Paul F. Kerr for the International Agricultural Corporation, their conclusions were in part as follows:

> Comparisons of X-ray diffraction patterns show that the phosphate constituents in the various phosphate rocks (Tennessee and Florida) are apparently in the form of a complex mineral of the apatite type, probably staffelite. This mineral contains carbonate and other chemical constituents peculiar to carbono-apatite and although complex, from the chemical standpoint, the mineral staffelite is present in all types of phosphate rock examined. Although shown by X-ray examination to be crystalline,

it is possible that microscopic examinations would show it to be so finely grained as to appear a solid jelly-like mass devoid of crystalline character.

This description of the phosphate rock probably explains why the finer portions, commonly known as "slimes," are so difficult to treat by flotation. This is true particularly of Florida pebble phosphate. There are many varieties of more coarsely crystalline apatite that do not form a near colloidal "slime" when ground.

Table 1 gives a complete screening analysis of a typical sample of "debris" as it comes from the dump. It requires about one cubic yard of this material to yield one long ton of ground and deslimed flotation feed.

History of Concentrator Operations

Early in 1926, Mr. John T. Burrows, vice president of the International Agricultural Corporation, in charge of phosphate rock operations, instigated an investigation as to possible methods of saving the fine rock then being wasted by the washers. James A. Barr, chief engineer, and Charles E. Heinrichs, of the Rock Department of the Corporation, were detailed to carry out this investigation.

After some experimental flotation work, it was discovered that Minerals Separation North American Corporation already owned at least one patent[1] covering the concentration of phosphate rock by flotation. That corporation was accordingly consulted and an agreement reached whereby the two companies would join forces in developing a commercial method of flotation concentration. This resulted in the formation of the Phosphate Recovery Corporation.

After a great deal of experimental and research work in the laboratories of both the Minerals Separation North American Corporation and the International Agricultural Corporation, a pilot plant was built in 1927 adjacent to the No. 11 washer of the last-named company, where current washer debris was available for experimental work. Many mechanical difficulties were encountered and a great many expensive changes made in equipment and finally a fairly satisfactory method worked out. During this time and up to the present, an active research program was carried out in the Phosphate Recovery Corporation research laboratory in Mulberry with a view to lowering costs and bettering metallurgy. At present six men are engaged in research and development work at the laboratory. This work has resulted in many improvements in the method of procedure. Ideas and methods developed by the laboratory have resulted in large savings in the commercial operations.

In January, 1929, the present Phosphate Recovery Corporation plant No. 1 was completed and commenced operations. During the pilot plant operation reagent cost was a considerable item but this was gradually reduced by desliming prior to flotation and the use of fuel oil or other petroleum oil in conjunction with fatty or soap-forming acids. Details of reagent addition also had a considerable bearing on the quantity of reagents necessary. It was found that the reagents for greatest efficiency should be added separately to a thick pulp, and then diluted before actual flotation.

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[1] Broadbridge and Edser: U. S. Patent No. 1547732 July 28. 1925.

After some experience in the operation of Phosphate Recovery Corporation plant No. 1, plans for a new plant having a capacity of about 2440 long tons of feed per day were drawn up and this plant was put into operation in April, 1930.

Plant No. 2, instead of utilizing current washer debris, was supplied with feed from several accumulated debris dumps adjacent to the plant. Although this plant has been in operation only a little over two years, many improvements have also been made in this period. During the early months of operations tailings at this plant averaged 8 to 12 per cent bone phosphate of lime and the grade of concentrate 70 to 72 per cent, whereas in recent months the average tailings have been 4 to 5 per cent B.P.L. and the grade of concentrate 74 to 76 per cent. Most of this improvement has been obtained by preparing the flotation feed so that as little plus 35-mesh material as possible is present. The plant as first designed had no grinding equipment whatever, but eight No. 6 Wilfley tables were installed to treat plus 20-mesh material. This form of table concentration is not gravity concentration, but is really a stratifying classification of suspended phosphate material in the form of floccules. The plus 20-mesh material is treated with reagents just as flotation feed is treated and the concentrate is carried transversely over the riffles while the quartz particles are carried along the riffles by the motion of the tables and discharged at the end of the table. It was found that, although this table separation was efficient if not over one ton per hour was fed to each table, better work might be done on the same feed if it were ground to the proper size and then handled in the regular flotation machines. To test out this method of separation as against the use of tables a 4 by 10-ft. rod mill was installed in May, 1931, and part of the oversize passed through this rod mill and thence to flotation. Much better over-all results and better costs were obtained by the combination of grinding and flotation.

The No. 2 plant, having a capacity of 100 long tons of feed per hour, originally cost nearly $200,000, and it is estimated that the new 60-ton per hour plant can be erected for about $80,000. This new plant cost is then less than $50 per short ton per day capacity as against usual flotation plant costs of $400 to $1000 per ton per day. Cheap plants and low operating costs have been made necessary because of the low value of the finished product and because of the fact that the flotation product must still compete in cost with a pebble product produced by washers.

Fig. 1 gives the present flow sheet of plant 2 and Fig. 2 shows a proposed flow sheet of an improved design of plant of about one-half the capacity of the present No. 2 plant. Changes that would be made in

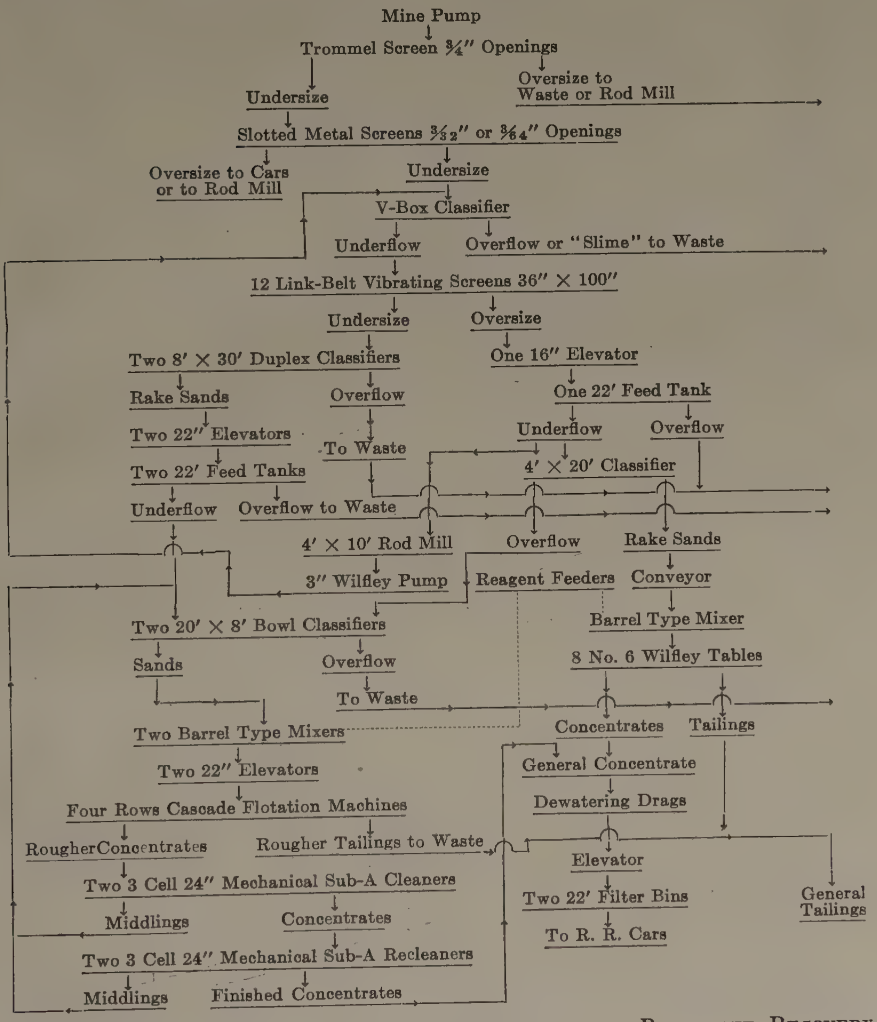

Fig. 1.—Flow sheet of No. 2 Plant, with rod mill, Phosphate Recovery Corporation.

building a new plant are obvious from a comparison of the flow sheets.

Present Methods of Concentration

At present the debris is pumped at 15 to 20 per cent solids from the pit to a point about 30 ft. above ground level, where it first passes through a revolving trommel screen with ⅞-in. circular openings. The oversize (mostly mud balls) from this trommel may be either rejected or laundered to the rod mill. The undersize from the trommel screen passes over a series of sloping slotted metal screens with openings 1 mm. wide. This screen oversize goes to the rod mill and the screen undersize to the V-box. The V-box is a V-shaped or U-shaped steel tank equipped with an inside overflow launder where part of the slimy water is removed to waste. On the bottom of the V-box are ten 4-in. plug valves through which the partially thickened product is drawn. The V-box underflow passes over twelve 3 by 8 ft. Link-Belt vibrating screens. Rectangular screen cloth of 24 mesh is usually used here. The screen oversize goes to a 16-in. bucket elevator discharging into a 22-ft. diameter cone-bottom steel bin. The vibrating screen undersize goes to two Dorr 8-ft. duplex classifiers, the overflow from these classifiers going to waste and the rake products discharging into two 22-in. bucket elevators that discharge into two 22-ft. diameter cone-bottom steel bins. Each of the

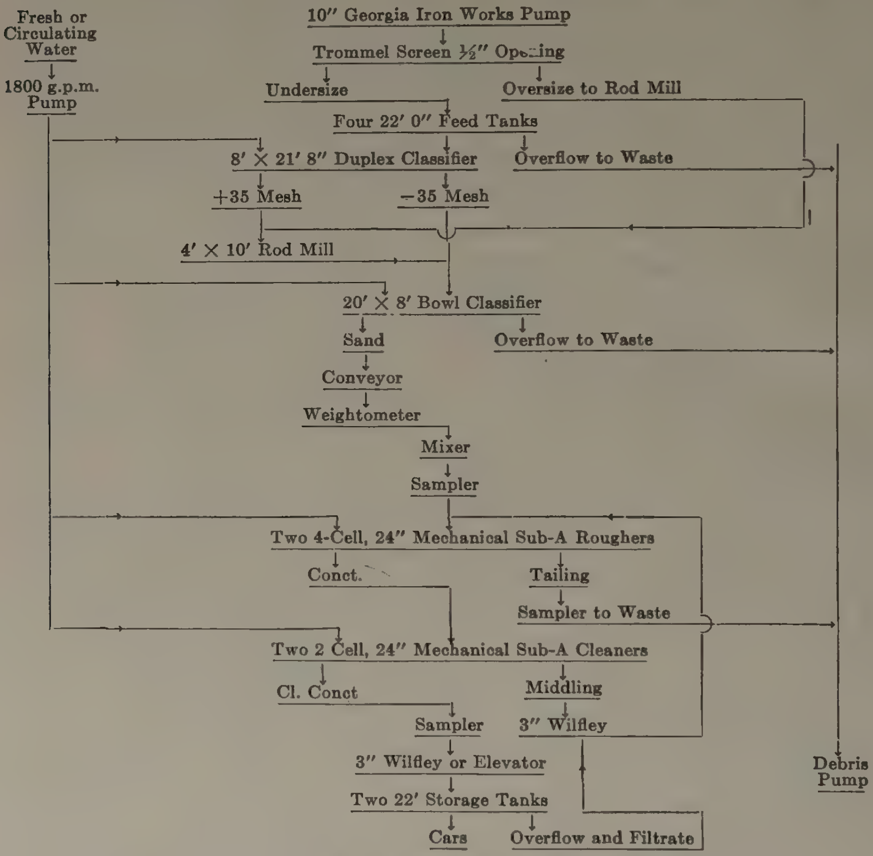

Fig. 2.—Proposed new flow sheet, 60 long tons feed per hour.

three bins has a storage capacity of about 300 tons. The duplex classifiers are operated to give approximately a 150-mesh separation, rejecting all material much finer than this, including most of the clay and other very fine suspended material.

The loss of very fine material in the several overflows (Fig. 1) varies greatly with the material treated, but does not amount to any considerable portion of the total phosphate material in the original feed. Investigations are now being carried on with a view to cutting down losses in this respect.

The rock from the coarse-rock bin may be treated in two ways. It may be drawn to the 4 by 10-ft. rod mill, and thence returned by 3-in. Wilfley pump to the V-box in closed circuit with the vibrating screens, or it may be drawn to a 4-ft. Dorr simplex classifier, sands from which are conveyed on an 18-in. belt up to a barrel-type mixer where reagents are added prior to table concentration. Overflow from the simplex classifier is laundered to join the fine bin underflow to two 20 by 8-ft. Dorr bowl classifiers.

At the present time the simplex classifier and table plant are used only when the rod mill has not sufficient capacity to grind all the coarse material to screen size. When an excess of coarse material is being mined, part of the coarse bin discharge goes to the rod mill and part to the simplex classifier and thence to the tables.

The discharge from the two fine-feed storage bins goes direct to 20 by 8-ft. Dorr bowl classifiers where the flotation feed receives a final washing and desliming. The overflow from the two bowls goes to waste and the rake product passes on to two cylindrical reagent mixers, each 16 ft. long by 5 ft. in diameter.

The flotation agents are added to the bowl rake product. Sufficient alkali is used to give a slight alkalinity to the pulp. The other agents are a fatty acid, a collector and a small proportion of a strong frother. Other reagents have been used successfully but the present combination has, to date, given the lowest cost. With the accumulation of experience, the total reagent consumption has been brought lower and lower, until now, on the average feed, it is less than 1½ lb. to the ton of feed.

The reagent mixers are rotating cylinders set at a slope of 3° 40′ and equipped with interior baffles for agitating the feed with the reagents. They discharge into the boots of two 22-in. bucket elevators, which discharge into the cascade flotation machine headers. Dilution water is added to the elevator discharge to give approximately 25 per cent solids in the first cascade cells. In the reagent mixers the solids average 60 to 70 per cent. There are four rows of cascade flotation machines, each row consisting of one 10 by 3-ft. cell and five 8 by 3-ft. cells in series. The rougher concentrate from the cascade machines goes to two three-cell 24-in. mechanical subaeration machines. The primary cleaner concentrate passes on to two more three-cell mechanical subaeration machines for a second cleaning. Cleaner tailings or middlings from both operations are pumped back to the 20-ft. bowl classifiers by a 3-in. and a 4-in. Wilfley pump.

In the light of present knowledge, we believe that mechanical subaeration machines will give better results as roughers than the cascade machines, and mechanical subaeration roughers are, therefore, used in the new design.

The regular mechanical subaeration design was changed somewhat on the machines used in phosphate flotation. Instead of a single spitz, these machines are double spitz and discharge at both sides, weirs being located at the center of the dividing compartment between the cells. This was done because of the low ratio of concentration and the very rapid float obtained on this type of feed. Air for the twelve mechanical subaeration cleaner cells is furnished by an Allen type A centrifugal blower, delivering 600 cu. ft. of air per minute at 1.25 lb. per sq. in. pressure. It is direct connected to a 7½-hp. motor of 3600 r.p.m. speed.

The secondary cleaner concentrate or finished product goes to two chain and blade type dewatering drag classifiers and the rake product from these dewatering drags discharges into one 22-in. bucket elevator. The bucket elevator discharges into either of two concentrate storage bins. These bins are of the same size and style as the feed storage bins except that they are equipped with four vertical filter panels covered with one layer of coarse screen cloth and one layer of fine screen cloth. The water drains readily from the concentrate through these filter panels and the partly dewatered concentrate is loaded through a sliding gate into filter-bottom gondola cars. By the time the cars are delivered at Prairie the average moisture content is about 13 per cent. The cars are weighed on track scales at Prairie and the moisture sample taken by hand as they are dumped. The dry weight of concentrate thus determined is used as the basis of mill tonnage calculations.

The Phosphate Recovery Corporation does not handle the drying of the concentrate; this is done by the International Agricultural Corporation, before shipment, in two 6 by 60-ft. Allis-Chalmers rotary kiln-type, oil-fired driers. Details of this process and equipment are given in the article referred to in *Pit and Quarry*, Dec. 30, 1931.

Waste products, including the rougher tailing from the cascade machines, overflow of the bowl classifiers and the overflow of the duplex classifiers, are pumped to a tailings pond by one 10-in. and one 8-in. Georgia Iron Works sand pump. The coarser material in the waste product settles out close to the pump discharge and the water passes on through a series of mined-out pits and canals until most of the suspended matter is settled out. It is then returned for re-use in the plant. Mined-out debris pits are used for tailings disposal as far as possible.

CONVEYORS, ELEVATORS AND DRIVES

Only one conveyor is used in the No. 2 plant, delivering table feed from the simplex classifier to the reagent mixer. This belt is 18 in. wide, 115 ft. between pulley centers and is inclined at 14°. It is driven by a 10-hp., 220-volt, 1150-r.p.m. motor, through speed reducer and sprocket and chain drive. Belt speed is 212 ft. per minute.

All elevators are set at 30° from vertical in order to assist dumping, and all pulleys are 59 ft. apart center to center. High-pressure sprays are also used in dumping the elevators as the feed handled has a strong tendency to remain in the buckets. The four 22-in. elevators handling fine feed and oiled feed are equipped with 18 by 10 by 10½-in. buckets, spaced 24-in. centers, and are driven 320 ft. per minute. Drives are 25-hp., 220-volt, 1150-r.p.m. motors, connected through enclosed gear-reduction units and sprocket and chain. The sprocket and chain was used in addition to gear reducers so that belt speed might be more easily changed, because it was known that this material is difficult to dump from buckets. The 22-in. concentrate elevator is the same as the feed elevators, except that it is driven at a slower speed of 164 ft. per minute. Pulleys for the 22-in. belts have 24-in. faces and are 36 in. in diameter. All are lagged with belting to prevent sand wear.

The coarse rock elevator is 16 in. wide, equipped with 14 by 8 by 8-in. buckets, spaced 24-in. centers. It is driven by a 10-hp., 220-volt, 1150-r.p.m. motor, through gear-reduction unit and sprockets at 320 ft. per minute.

The Link-Belt vibrating screens are individually driven by a 2-hp., 220-volt, 1720-r.p.m. motor through V-belt drive. Duplex and bowl classifier rakes are driven at 27 strokes per minute, by 10-hp., 220-volt, 860-r.p.m. motors through V-rope drives. The bowl rakes are driven at 3.25 r.p.m. by 5-hp., 220-volt, 1140-r.p.m. motors through V-rope and worm gear.

The 4 by 10-ft. rod mill is driven at 28 r.p.m. by a 75-hp., 2300-volt, 870-r.p.m. motor. The motor is connected by 12 V-ropes to the pinion shaft.

The 5 by 16-ft. flotation reagent mixers are driven at 14 to 16 r.p.m. by 15-hp., 220-volt, 1150-r.p.m. motors through V-rope, sprocket and chain and pinion drives to a ring gear. They are mounted on rollers at a slope of 3° 40′ and were especially designed for this purpose. The table feed reagent mixer is 30 in. in diameter by 16 ft. long and is similarly mounted and driven by a 7½-hp., 220-volt, 1140-r.p.m. motor at 16 r.p.m., through speed reducer and sprocket and chain.

Each of the No. 6 Wilfley tables is driven by a 1½-hp., 220-volt, 1165-r.p.m. motor through V-ropes.

The mechanical subaeration flotation machines are divided into two six-cell gear-type units, each driven by a 40-hp., 220-volt, 860-r.p.m. motor, through V-ropes, impeller speed being 345 r.p.m.

The concentrate dewatering drags are driven by 5-hp., 220-volt, 1140-r.p.m. motors through sprocket and chain and gear reducers.

The 4-in. Wilfley middling pump is driven by a 30-hp., 220-volt, 1165-r.p.m. motor, direct connected and the 3-in. Wilfley middling pump by a 15-hp., 220-volt, 1160-r.p.m. motor also direct connected.

The 3-in. Wilfley pump used to return rod-mill discharge to the V-box is driven by a 15-hp., 220-volt, 1160-r.p.m. motor, direct connected.

The 8-in. Georgia Iron Works tailings pump is driven by a 100-hp., 2300-volt variable-speed slip-ring motor direct connected, and the 10-in. Georgia Iron Works tailings pump by a 150-hp., 2300-volt variable-speed slip-ring motor, also direct connected.

The entire mill-water supply is furnished by one low-pressure and one high-pressure pump. The low-pressure pump is 12 by 10 size, delivers 5000 gal. per min. at 100-ft. head, and is direct connected to a 150-hp., 2300-volt, 1165-r.p.m. motor. The high-pressure pump is 3 by 2½ size, delivers 225 gal. per min. at 230 ft. head and is direct connected to a 30-hp., 220-volt, 1740-r.p.m. motor.

Flotation reagents are stored in tanks at ground level and are pumped to feeders at about 30 ft. elevation by a four-cylinder plunger pump. Feeders are of the cup and disk type, with a swinging adjustable drip tray. Adjustments are made by varying the number of cups and moving the drip tray up or down. The feeders are driven by 1-hp. motors through worm gear-reduction units.

Sampling

No adequate provision for automatic samples was made in the original design of the plant, but a Geary-Jennings automatic electric sampler has since been installed to sample the general mill tailing. This cutter discharges into a duck "cradle" where most of the water drains off between cuts.

Concentrate is sampled by a water-dump cutter as it is delivered to the filter bins. Feed samples and intermediate samples of table and flotation products are taken by hand at 30-min. intervals. All samples are collected, dried in an electric oven and prepared for assay at the plant at the end of each 8-hr. shift.

All analytical work is done by the International Agricultural Corporation analytical laboratory.

Water Supply

As already stated, most of the water used in flotation is returned and re-used, and this is augmented by rainfall, since the average annual rainfall here is about 60 in. The International Agricultural Corporation has several deep wells in the vicinity, however, and in these the water rises to about 30 ft. below ground level. This water is pumped into the circulating system as needed. During long periods of drought the use of larger quantities of this water causes increased reagent consumption, because it is "hard." The circulation water is thoroughly softened by the reagents, but with hard water the reagents form a calcium soap. If much deep-well water were used, it would no doubt pay to soften it prior to use in flotation. Total water used amounts to 5225 gal. per min. or about 2.030 gal. per min. per short ton milled per day. This figure is high, but this is due to the fact that a great deal of water is sent to waste in the "desliming" operation, by the duplex and bowl classifiers, a condition not usual in other flotation plants.

Concentrator Recovery

The month of December, 1931, was chosen as typical for metallurgy and costs, but much lower grade of feed has been handled since then, and metallurgical results are also given for July and August, 1932. In spite of further curtailment during these later months, costs per ton of feed were maintained at about the same figure as for December, 1931. If the plant had been operated full time during July and August, 1932, costs could have been reduced to about 11¢ per short ton of feed. The No. 2 plant has frequently handled 57,000 to 60,000 tons of feed per month, but December was chosen as being average. The tonnages shown for December were made in 24 operating days, the plant being shut down on Sundays and three additional days at Christmas time.

In the fiscal year ending June 30, 1931, this plant shipped 222,607 long tons of concentrate and in the following 12 months shipped 123,383 long tons. The reduction was partly due to curtailment and partly to lower grade of feed handled during the second period.

Table 2 shows metallurgical results for the months of December, 1931, July and August, 1932. Table 3 gives other metallurgical data for December, 1931. Table 4 gives actual screening analyses of monthly composite general mill tailings and concentrate, and a calculated feed screen analysis for December, 1931. Mesh recoveries are also shown. This and other screening analyses show that all the feed should be ground to pass 35 mesh and remain on 100 mesh as far as possible. Equipment at the No. 2 plant is not capable of making this fine a separation. The vibrating screens would not have sufficient tonnage capacity if 28 to 35-mesh screens were used, and one 4 by 10-ft. rod mill has not the grinding capacity necessary for this tonnage. In the new plant design, screens are eliminated and the 35-mesh separation made by classification.

The low grade of the finer sizes in the concentrate does not necessarily indicate that the concentrate would be lower if all the feed were ground to this mesh. With very little difference in floatability between the two predominant minerals (quartz and phosphate), the plant operator applies reagents to float all possible coarse phosphate (plus 35-mesh material), and this condition will inevitably float a good deal of 100 to 150-mesh quartz. If all the phosphate grains were minus 35-mesh or smaller, the separation could be made more sharp, even in the 100 and 150-mesh range. It is a decided advantage, however, to keep most of the feed particles in the 48 to 65-mesh range, for at these sizes the reagent consumption is lower and the float much more rapid than with finer material.

TABLE 2.—*Phosphate Recovery Corporation Plant No. 2. Average Monthly Mill Assays and Recoveries*

Product	Long Tons	Bone Phosphate of Lime, Per Cent	Bone Phosphate of Lime, Recovery, Per Cent	Concentration Ratio
December, 1931				
Feed	52,323.38	21.374		
Concentrates	12,271.00	75.049	82.348	4.2639
Tailing	40,052.38	4.929		
July, 1932				
Feed	43,265.81	18.214		
Concentrates	8,566.00	74.093	80.537	5.0509
Tailing	34,699.81	4.420		
August, 1932				
Feed	50,859.87	17.786		
Concentrates	9,570.00	74.250	78.552	5.3145
Tailing	41,289.87	4.699		

TABLE 3.—*Metallurgical Data. Concentrator No. 2, Phosphate Recovery Corporation*

PERIOD COVERED, DECEMBER, 1931

Total long tons ore treated[a]	52,323.38	Total average tons concentrate per 24 hr.	538.91
Days operated (actual)[b]	22.77	Recovery of bone phosphate of lime	82.348
Hours operated per day	24 (3 shifts)	Ratio of concentration	4.2639
Average tonnage per 24 hr.	2,297.91		
Total tons concentrate	12,271.00		

[a] All tonnages are long tons of 2240 lb.

[b] Plant shut down seven days (three days Christmas holidays and four Sundays due to production curtailment). Balance of lost time due to shutdown for repairs and accidental causes.

TABLE 4.—*No. 2 General Mill Monthly Composites for December, 1931, Phosphate Recovery Corporation*

Mesh	Weight, Per Cent	Weight, Cumulative Per Cent	Bone Phosphate of Lime, Per Cent	Insoluble	Recovery, Bone Phosphate of Lime, Per Cent
CALCULATED FEED					
Total	100.00		21.37		
+ 20	Trace				
+ 28	4.77	4.77	50.32		
+ 35	12.97	17.74	35.78		
+ 48	25.59	43.33	20.36		
+ 65	31.60	74.93	17.12		
+100	18.26	93.19	15.77		
+150	6.08	99.27	11.68		
−150	0.73	100.00	16.44		
ACTUAL MILL CONCENTRATES					
Total	100.00		75.05	7.92	82.4
+20	Trace				
28	6.29	6 29	78.56	3.95	47.9
35	18.54	24 83	79.07	3.58	74.1
48	24.83	49 66	78.37	4.73	87.5
65	28.81	78 47	74.03	8.67	92.6
100	16.23	94 70	70.29	12.81	93.1
150	4.31	99 01	64.63	19.88	91.6
−150	0.99	100 00	47.17	40.01	91.7
ACTUAL MILL TAILING					
Total	100.00		4.93		
+20	Trace				
28	4.31	4.31	38.03		
35	11.26	15.57	13.90		
48	25.83	41.40	3.28		
65	32.45	73.85	1.59		
100	18.87	92.72	1.39		
150	6.62	99.34	1.20		
−150	0.66	100.00	2.66		

SUMMARY OF COSTS

Table 5 gives the details of operating costs, but such costs on an operation of this size are not kept by departments. Power is metered at the plant, but is not metered by departments. Total power used in December, 1931, for milling purposes was 5.344 kw-hr. per short ton of feed handled. The total actual load at the plant averages about 770 hp., including water supply. Total rated motor horsepower is 920, some of the motors being operated intermittently and some at less than capacity.

TABLE 5.—*Summary of Direct Operating Costs, No. 2 Flotation Concentrator, Phosphate Recovery Corporation, for the Month of December,* 1931

Long Tons Ore Treated, 52,323.38 Long Tons Concentrates Produced, 12,271.00
Short Tons Ore Treated, 58,602.19 Short Tons Concentrates Produced, 13,743.52

	Total for Month	Per Long Ton Feed	Per Short Ton Feed
Operating labor	$1,268.19	$0.0242	$0.0217
Power	3,131.80	0.0599	0.0534
Supplies (operating)	133.18	0.0025	0.0023
Reagents	1,245.03	0.0238	0.0212
Repair labor	329.74	0.0063	0.0056
Repair supplies	614.08	0.0117	0.0105
Sampling	129.01	0.0025	0.0022
Assaying	138.95	0.0027	0.0024
Superintendent, office and local overhead	755.07	0.0144	0.0129
Total	$7,745.05	$0.1480	$0.1322
SEGREGATED INTO LABOR, SUPPLIES AND POWER			
Total labor	$1,726.95	$0.0330	$0.0295
Power	3,131.80	0.0599	0.0534
Reagents	1,245.03	0.0238	0.0212
Supplies	747.26	0.0143	0.0128
Superintendent, office and local overhead[a]	894.02	0.0170	0.0153
Total	$7,745.05	$0.1480	$0.1322

[a] Including assaying.

APPENDIX.—NEW DEVELOPMENTS IN THE FLOTATION CONCENTRATION OF PHOSPHATE ROCK*

Since the preceding article was written, the flotation process for concentration of the finer fractions of phosphate rock has developed rapidly. The Phosphate Recovery Corporation has licensed several companies in Florida and Tennessee to use the processes covered by various patents and several new plants are either in operation or under construction.

The No. 1 plant of the Phosphate Recovery Corporation has been dismantled and most of the machinery moved to Pembroke, Florida, 10 miles south of Bartow, Polk County, where a new 50-ton-per-hour plant is now in operation. It is expected that this flotation plant will produce about 150,000 tons per year of high-grade concentrate. Feed will be obtained from several dumps of accumulated washer waste that were produced some years ago. Extensive prospecting and laboratory test work indicate that there is sufficient material available to

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* Manuscript received at the office of the Institute Nov. 20, 1934.

operate this concentrator at the above rate for seven to nine years. This particular dump of washer waste is one of the few older ones in the Florida pebble-rock field where overburden was not mixed with washer waste at the time. This early segregation of barren material from valuable low-grade fines was due undoubtedly to the foresight of Mr. S. D. Gooch and others in the management of the Coronet Phosphate Co., the company that originally owned and mined this area.

The flow sheet of the Pembroke flotation plant (known as Phosphate Recovery Corporation plant No. 4) is shown in Fig. 3. Instead of mining with hydraulic guns, a suction dredge will be used. This method is used for mining concrete sand at Lake Wales, Florida, and has proved successful. Since the bottom of the dump material is about 20 ft. below normal water level, a saving in pumping head can be effected as against keeping the pit pumped out as the mining goes on. No hydraulic water, except possibly a small high-pressure jet, will be necessary and this also will make the dredge operation more economical.

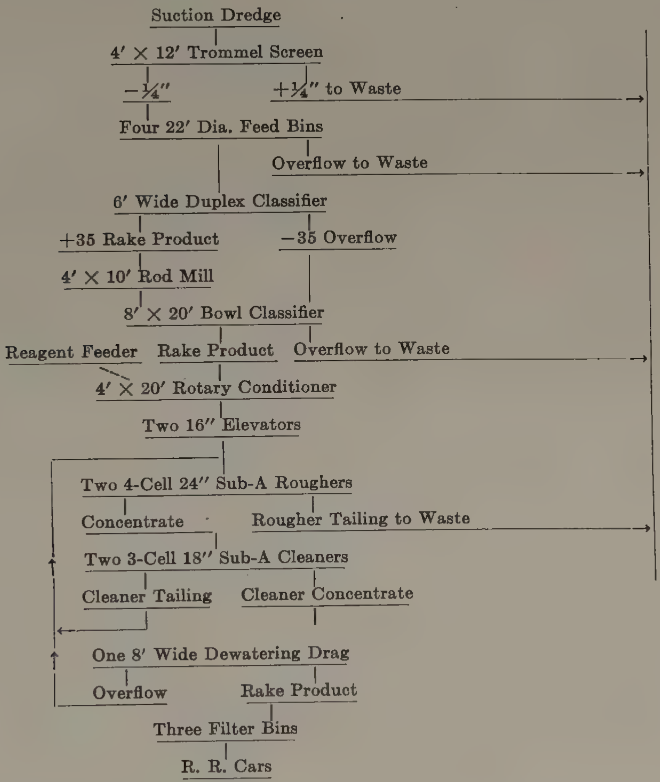

FIG. 3.—FLOW SHEET OF PEMBROKE FLOTATION PLANT, 50 LONG TONS FEED PER HOUR.

The dredge is a rectangular scow, 40 by 20 ft., on which is mounted a 10-in. Georgia Iron Works pump, direct connected to a 150-hp. variable-

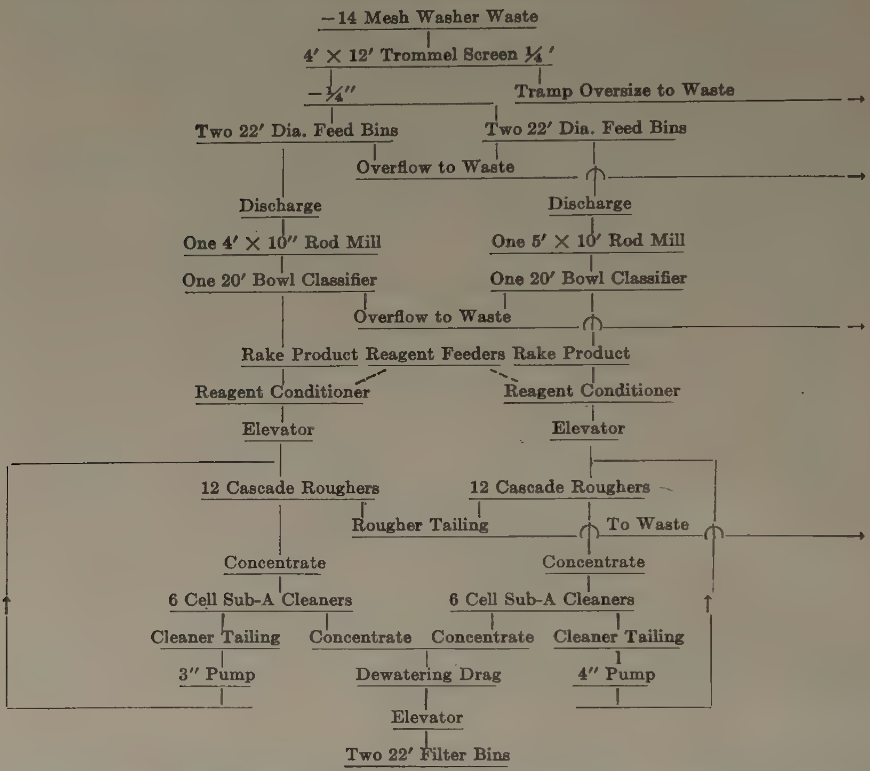

FIG. 4.—REVISED FLOW SHEET OF PHOSPHATE RECOVERY CORPORATION NO. 2 PLANT, 80 TO 100 LONG TONS FEED PER HOUR.

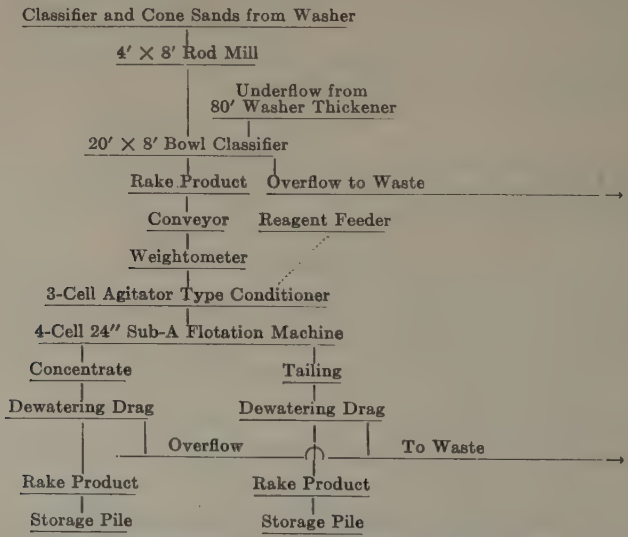

FIG. 5.—FLOW SHEET AT WALES, TENNESSEE, 15 TO 25 LONG TONS FEED PER HOUR.

speed motor. The pump suction is mounted on a swinging A-frame so that it can be raised, lowered or swung from side to side. A motor-driven winch is used for these operations. Pumping at the rate of 175 to 200 cu. yd. of dump material per hour, this dredge will be operated 6 to 10 hr. per day to fill the flotation-feed bins. The flotation plant will then draw on the feed stored in the bins for the remaining 18 to 14 hr. per day.

TABLE 6.—*Metallurgical Results and Costs for Year Ending June 30, 1934, P. R. C. Plant No.* 1

METALLURGICAL RESULTS

	Long Tons	Bone Phosphate of Lime		Ratio Concentration
		Per Cent	Per Cent Recovery	
Feed	137152	26.402		
Concentrate	40716	73.634	82.796	3.368
Tailing	96436	6.460		
Total possible operating hours				4416
Actual operating hours				2906
Percentage of time actually operating				65.82

COSTS

	Per Short Ton Feed	Per Long Ton Concentrate
Operating labor	$0.0369	$0.1391
Power	0.0599	0.2260
Reagents	0.0387	0.1461
Repair labor and supplies	0.0149	0.0562
Sampling and assaying	0.0052	0.0196
Superintendence and overhead	0.0129	0.0488
Total direct operating cost	$0.1685	$0.6358

The flow sheet at Pembroke follows closely the proposed new flow sheet (Fig. 2) and is very much simpler than the original flow sheet at plant No. 2 (Fig. 1).

Since the previous article was written the flow sheet at plant No. 2, near Mulberry, Florida, has also been simplified. This plant is now handling current minus 14-mesh waste from the No. 11 washer of International Agricultural Corporation. After preliminary dewatering in a wooden V-box at the washer, the waste is pumped about 2000 ft. to four feed bins, each 22 ft. in diameter, one new bin having been recently installed. The revised flow sheet at No. 2 plant is shown in Fig. 4.

The Phosphate Recovery Corporation is also operating a third flotation plant in conjunction with the International Agricultural Corporation washer at Wales, Tenn. At Wales, the entire washer output is used as feed for the flotation plant, following the flow sheet shown in Fig. 5.

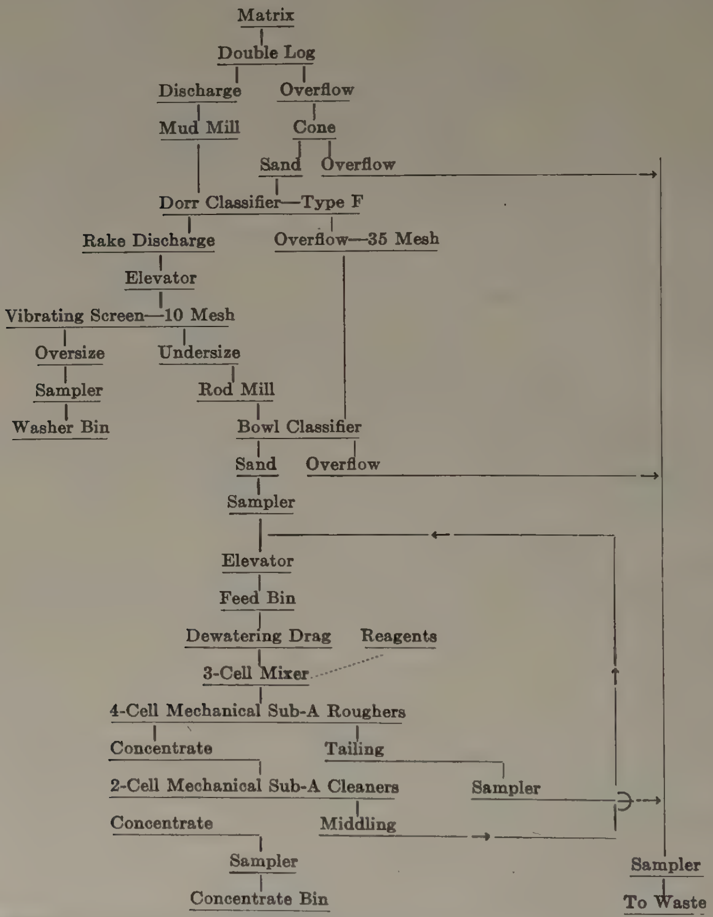

FIG. 6.—FLOW SHEET OF LABORATORY WASHER AND FLOTATION UNIT, PHOSPHATE RECOVERY CORPORATION.

Metallurgical results and direct costs for the year ending June 30, 1934, at the No. 1 plant are shown in Table 6.

The Phosphate Recovery Corporation research and experimental laboratory at Mulberry has handled hundreds of samples of dump material and matrix for other companies during the past few years, giving these companies information as to probable results on the samples at the washer and flotation plant. During the past year, a continuous laboratory washing and flotation unit was installed at the laboratory. The washer unit consists of a small log washer, cylindrical scrubber and vibrating screen. The screen undersize is run continuously to a flotation unit consisting of two small classifiers, settling cone, rod mill, feed-storage bins, reagent conditioner and seven-cell 4-in. mechanical flotation machine. The entire unit handles 300 to 600 lb. of matrix per hour, and a sample of about 2 tons is required for an entire day's run. Several such samples have been handled and the results used to compare with larger commercial operation. In at least one instance such a test run showed that the commercial operations were not efficient and this led to an entirely new washer flow sheet at that deposit. A diagrammatic flow sheet of the laboratory washer and flotation unit is shown in Fig. 6.

Relative Floatability of Silicate Minerals

By John Mark Patek,* Member A. I. M. E.

(New York Meeting, February, 1934)

Knowledge of the relative floatability of silicate minerals is increasing in importance as flotation is being applied to the concentration of nonsulfides. Many silicates are in themselves commercial products amenable to concentration by flotation. Moreover, the selective flotation of other nonsulfide minerals, such as cassiterite, depends largely upon the floatability of associated gangue silicates. Differences in floatability existing between the various silicates have not been sufficiently appreciated; for example, the feldspars that differ greatly in floatability have generally been merely treated as "feldspar." It was the purpose of the work described in this paper to determine by experiment the differences in floatability between silicates; to investigate the causes for such differences, and to discover a means of predetermining approximate floatability without the necessity of experimentation.

Determining Floatability

The relative floatability of 20 silicates so selected as to represent all of the more common silicate types[1] was obtained by flotation tests enumerated in Table 1.

The mineral to be floated was crushed dry on a steel plate, and screened repeatedly during crushing through an 80-mesh screen in order to produce a sand with all grains as close to 80 mesh as possible. The sand was placed in dilute (6N) hydrochloric acid until free of soluble foreign material, and then washed with tap water about ten times in order to remove both acid and fines. The resultant product, which averaged 82 per cent plus 200 mesh, was floated in 50-gram charges in a 50-gram Denver celluloid flotation machine with 0.008 gram of terpineol, and from 0.01 to 0.05 gram of oleic acid in a solution acidified with sulfuric acid.

The degree of separation of one mineral from another was determined by counting under a microscope the grains of each mineral, both in the concentrate and in the tailings, and comparing their relative abundance in each. Unless the comparison showed a decided difference in amount,

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* Mining Engineer and Geologist, Milwaukee, Wis.

[1] Dana and Ford: Dana's Textbook of Mineralogy. New York, 1933. John Wiley and Sons.

the flotation test was repeated with varying proportions of reagents until a difference was produced, or until the minerals were known to be approximately equally floatable, as are quartz and orthoclase feldspar.

TABLE 1.—*List of Minerals in Order of Floatability, and Tests That Furnished the Data*

Order of Floatability	Mineral
1	Quartz Orthoclase
2	Albite
3	Nephelite
4	Muscovite Labradorite
5	Biotite
6	Spodumene
7	Anorthite
8	Augite
9	Rhodonite
10	Titanite
11	Tremolite
12	Epidote
13	Cyanite
14	Olivine
15	Topaz
16	Almandite
17	Tourmaline (black) Zircon

Individual Flotation Tests
1. Muscovite Labradorite Biotite
2. Spodumene Olivine
3. Biotite Spodumene Anorthite
4. Anorthite Tourmaline
5. Augite Olivine
6. Rhodonite Epidote Cyanite
7. Olivine Almandite
8. Spodumene Topaz
9. Olivine Cyanite
10. Epidote Olivine
11. Biotite Tremolite
12. Labradorite Biotite Tremolite
13. Augite Olivine Topaz Tourmaline
14. Titanite Tourmaline Almandite
15. Epidote Topaz Tourmaline
16. Anorthite Titanite Augite
17. Anorthite Titanite Augite Tremolite
18. Titanite Epidote Topaz Tourmaline
19. Titanite Tremolite Epidote Olivine
20. Rhodonite Titanite Cyanite Olivine
21. Titanite Epidote Cyanite
22. Augite Rhodonite Tremolite
23. Tourmaline Zircon
24. Augite Rhodonite Olivine Tourmaline
25. Quartz Orthoclase
26. Quartz Orthoclase Albite Muscovite
27. Albite Nephelite Muscovite

The relative floatability of the 20 silicates is shown in Table 1. The minerals are numbered from 1 to 17 in the ascending order of floatability. It should not be supposed that perfect separation was always obtained between two adjoining minerals, but good separation was made at approximately every third mineral; that is, between 1 and 4, 4 and 7, and so forth.

It has long been recognized that floatability varies with luster,[2] and increases approximately in the following order: vitreous, resinous, adamantine, metallic. A correlation between the numerical notation for luster, i.e., the refractive index, and density has also been known; it may be expressed[3] by $\frac{(n-1)}{d} = K$, where n = refractive index, d = density and K = a constant. Experiments to be described indicate that density bears an even closer relation to floatability than does refractive index, and that it may be correlated with what has been found to be the principal factor influencing floatability of silicate minerals; namely, the natural wettability of the mineral.

WETTABILITY

Liquid on a plane surface will spread or contract, depending on how well the liquid wets the solid.[4] When a drop of liquid spreads upon a solid surface, the angle between the surface of the liquid and that of the solid decreases until spreading slows down and finally ceases. There results a definite equilibrium angle between the solid and liquid surfaces; it is characteristic of the particular gas, liquid, and solid surfaces involved,[5] and is known as the contact angle. In this paper, the tendency of a liquid to spread or contract at a mineral surface will be viewed as a mineral property and will be referred to as either "mineral wettability" or "mineral unwettability."

Wettability[6] varies with the energy of adhesion between liquid and solid, the energy of cohesion of the liquid, and the surface tension at the solid-liquid interface. It may be expressed in terms of the contact angle Θ.

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[2] A. F. Taggart: A Manual of Flotation Processes. New York, 1921. John Wiley and Sons.

[3] Formula of Gladstone and Dale: U. S. Geol. Survey *Bull.* 679 (1921) 30. Formula is applicable to silicates.

[4] E. L. McMillen: *Ind. & Eng. Chem.* (1930) **22,** 890–893.

[5] W. D. Harkins and R. Dahlstrom: *Ind. & Eng. Chem.* (1930) **22,** 897–903.

[6] H. Freundlich: *Trans.* Electrochem. Soc. (1931) **60,** 389–390.

A. F. Taggart: Handbook of Ore Dressing, 780. New York, 1927. John Wiley and Sons.

E. K. Rideal: Surface Chemistry, 5, 6, 256. Cambridge, 1930. University Press.

H. L. Sulman: *Trans.* Inst. Min. and Met. (1919) **29,** 44–204.

A. F. Taggart, T. C. Taylor and C. R. Ince: *Trans.* A.I.M.E. (1930) **87,** 297.

A. M. Gaudin: Flotation, 343. New York, 1932. McGraw-Hill Book Co. Interfacial tension is the "water-repelling property" at nonpolar surfaces.

A. W. Fahrenwald: *Trans.* Electrochem. Soc. (1931) **60,** 312.

When $\Theta = 0°$, spreading of the liquid continues indefinitely; when $\Theta = 0°$ to $90°$, spreading is only partial; and when $\Theta = 90°$ to $180°$, contraction rather than spreading occurs.

When the cohesion of the liquid is less than the adhesion between solid and liquid, $\Theta = 0°$. When the cohesion of the liquid is greater than the adhesion between solid and liquid, but when the solid-liquid interfacial tension is less than the solid-liquid adhesion, $\Theta = 0°$ to $90°$. When the liquid cohesion is greater than the solid-liquid adhesion, and the solid-liquid interfacial tension is greater than the solid-liquid adhesion, $\Theta = 90°$ to $180°$.

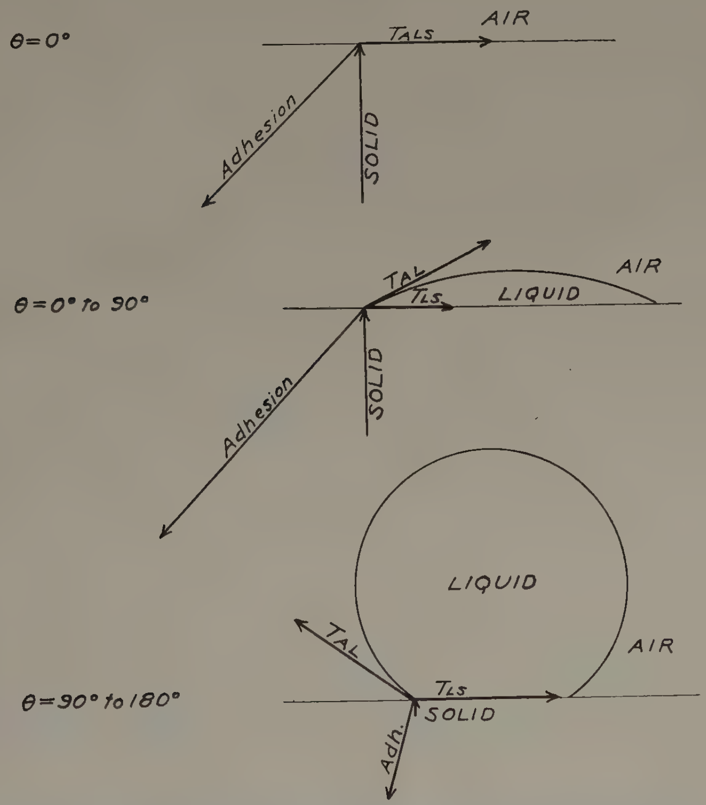

FIG. 1.—FORCE DIAGRAMS SHOWING RELATION OF LIQUID-SOLID ADHESION TO AIR-LIQUID AND LIQUID-SOLID INTERFACIAL SURFACE TENSIONS.

These relationships are expressed by force diagrams in Fig. 1. Both vertical and horizontal components are considered in these diagrams; the drop is not shown moving upward, as is frequently the case with representations of this type. The solid surface energy acts to cause adhesion between liquid and solid, and thereby produces spreading of the liquid on the solid. The force causing spreading is therefore represented as acting downward instead of along the solid surface.

The work of adhesion between a liquid and a solid is represented in terms of the liquid surface tension in the familiar equation of Young,[7]

$$Wa = \gamma_L\ (1 + \cos \Theta) \text{ ergs per centimeter}$$

where Wa = the work of adhesion, γ_L = the liquid, or air-liquid surface tension, and Θ = the contact angle. From this equation it follows that when Θ equals 180°, the work of adhesion is 0; i.e., adhesion does not exist. On the other hand, when Θ equals 0°, the work of adhesion equals $2\gamma_L$ ergs per centimeter which is the work of cohesion of any liquid. In other words, all of the attractive force of the liquid is employed in adhesion to the solid instead of in cohesion with itself.

Now, if Young's equation is altered to express the work of liquid cohesion instead of adhesion to another substance, it will read,

$$Wc = \gamma_L\ (1 - \cos \Theta) \text{ ergs per centimeter}$$

where Wc = the work of cohesion. This expression represents the tendency of the liquid at a mineral surface to cohere or contract, in which case it might be said to represent the crowding out or repulsive tendency of the liquid. Since the art of flotation is based on the elastic repulsion, which the liquid cohesion produces, the equation derived from that of Young will be used to express what will be termed "unwettability."[8] If the same liquid is employed in obtaining different values of Θ, γ_L becomes a constant, and unwettability (Wc) varies as $1 - \cos \Theta$.

Young and Harkins[9] state that in experiments in which the angle of contact Θ has been determined, the difficulty of cleaning the surfaces of the solids is so great that it is doubtful whether experiments have ever been conducted with pure solid surfaces. They further state that by the use of various optical methods it has been shown that, provided glass is already covered by a film of the liquid, Θ between glass and a number of liquids is zero; but when the glass has become dry, Θ may be greater than zero.

The writer has found this true of silicate and oxide minerals, and others have found it true of the sulfides. Dry mineral surfaces seem to show with water a contact angle of between 15° and 70°; in other words, the adhesion between liquid and solid is such that the spreading of a drop of liquid over the dry solid surface will be limited. If a clean mineral surface is submerged in water and then withdrawn, the surface will be completely filmed with water which adheres so strongly that it can be removed only by evaporation or wiping. When minus 200-mesh sand is wetted with water,[10] the energy of the water devoted to the adhesion between it and the sand is such that the water will show a lower freezing point, a lower rate of evaporation, and a lower vapor pressure. The energy of adhesion of a particular liquid depends upon the nature of the substance to which it adheres.[11] Thus the energy of adhesion between water and nonpolar hydrocarbons is lower than that between water and inorganic substances in general.

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[7] E. K. Rideal: Surface Chemistry, 5. Cambridge, 1930. University Press.

[8] O. C. Shepard: *Min. & Met.* (1932) **13**, 282.

I. W. Wark: *Trans.* Electrochem. Soc. (1932) **61**, 427.

[9] T. F. Young and W. D. Harkins: Int. Critical Tables, **4**, 434. New York, 1928. McGraw-Hill Book Co.

If a captive bubble of air were lowered against a clean mineral surface to which adheres a film of water, there probably would be no tendency for the water to contract and expose the mineral surface; consequently the contact angle between air-water and water-mineral interfaces would be 0°. If, however, the mineral is partly coated with an organic substance having a contact angle greater than 90°, or with a gas such as a sulfide might possess on its surface after exposure to air, the liquid will contract, and a contact angle will result.

In the captive-bubble test, the contact angle of a polished sulfide surface coated with hydrocarbon molecules varies with the chemical nature of the coating substance. Methyl xanthate, for example, gives an angle of about 50°, while that of cetyl xanthate is approximately 96°, the contact angle varying with the length of the hydrocarbon chain.[12] Of more interest as regards wettability is the effect of changing the orientation of the hydrocarbon chain. In the isomeric series of propyl, butyl and amyl xanthates, the promoter activity increases in the order: normal, iso, and secondary.[13] In a series of tests on Wisconsin sphalerite ore,[14] the normal butyl xanthate gave a zinc recovery of 45 per cent, while the secondary butyl gave a 70 per cent recovery. The nonpolar hydrocarbon group of the normal butyl xanthate is linked with its polar group at one end of the hydrocarbon chain, which probably would be at right angles to the mineral surface. On the other hand, in the secondary butyl xanthate, the polar group is attached near the middle of the hydrocarbon chain, and so the chain probably would lie somewhat parallel to the mineral surface; this should greatly increase the amount of unwettable area at the mineral surface.

If the captive bubble is applied without pressure sufficient to squeeze out or wipe aside the water film on the mineral surface, one might anticipate an angle of either 0° or plus 90°. However, if the mineral does not present to the captive bubble a complete front of slightly wettable nonpolar hydrocarbon, but rather a mixed field of wettable and unwettable points, the liquid might contract without producing a total contact angle of over 90°. The tendency of the liquid to contract at unwettable points is probably the cause of the extreme sensitivity of the captive-bubble test in detecting traces of organic matter.

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[10] F. W. Parker: *Jnl.* Amer. Chem. Soc. (1921) **43,** 1011–1017; *Soil Science* (1922) **8,** 43–54.

[11] Reference of footnote 5.

[12] I. W. Wark and A. B. Cox: Page 189, this volume.

[13] R. S. Bemis and E. J. Knechtges: Unpublished thesis on the Promoter Activity of Alkyl Xanthates, University of Wisconsin, 1933.

[14] Reference of footnote 13.

The adhesion between a liquid and a solid undoubtedly is due to chemical affinity, probably the tendency of the solid to dissolve in or combine with the liquid. For example, if mercury is placed in a series of cups composed of iron, sodium silicate glass, zinc and silver, the meniscus of the mercury in each cup will be somewhat as shown in Fig. 2.

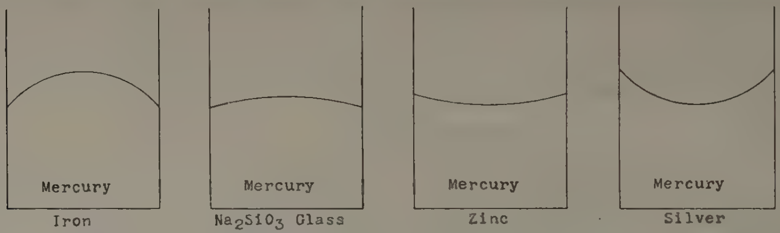

FIG. 2.—DIFFERENCES IN ATTRACTION OF LIQUID FOR SOLID SHOWN BY MENISCUS OF MERCURY IN CUPS COMPOSED OF VARIOUS SOLID SUBSTANCES.

This is because of the increasing solubility of the cup in mercury, in going from iron to silver.[15]

OBSERVATION OF CONTACT ANGLES AT MINERAL SURFACES

In order to determine the relative tendency of liquid to wet the surface of each of the 20 silicates (Table 1), contact-angle readings were taken with drops of liquid on dry, clean, flat, ground but unpolished mineral surfaces in the presence of air. This method makes possible the recording of the wettability of surfaces not previously filmed with either water, hydrocarbons, or the Beilby layer of polished surfaces.[16] It might be said that the minerals are subjected to the dust and grease in the air, but if there is contamination from that source it was not discovered, and for all practical purposes the minerals remain clean. Of course, sulfide minerals react with the oxygen of the air, but oxides do not.

There was no desire to establish absolute values for the contact angles, only comparative values. What was desired were angles definitely related to floatability. This required the use of mineral surfaces free of unsatisfied residual valences.[17] In flotation the presence of soluble salts would satisfy such valences. Cleaved surfaces, and especially those freshly cleaved, were wetted with unusual activity, probably because of residual valences, but ground surfaces and crystal faces were not so wetted.

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[15] Obtained in conversation from Louis Kahlenberg, University of Wisconsin.

[16] F. Osmond: The Microscopic Analysis of Metals, 85–89, 1924. Charles Griffin & Co. Ltd.

[17] I. Langmuir: *Jnl.* Amer. Chem. Soc. (1916–17) **38,** 2221–2295; **39,** 1848–1906.

To produce a satisfactory mineral surface, the following procedure was adopted: (1) The mineral was ground wet on a sandstone wheel to produce one flat face; (2) it was then placed in 6N hydrochloric acid for several minutes to dissolve foreign material; (3) it was cleaned several times with distilled water and dried; and (4) ether was used to remove any possible trace of hydrocarbon.

TABLE 2.—*Contact Angle, Comparative Unwettability* (1 − *cos* θ), *Specific Gravity and Refractive Index of Each of the Twenty Silicates*

Mineral	Contact Angles of Individual Drops	Average Contact Angle	1 − cos θ	Specific Gravity[a]	Calculated Specific Gravity	Average Refractive Index
Orthoclase	18°, 20, 20, 22	20°	0.060	2.57	2.60	1.522
Quartz	21, 21, 22, 21	21	0.066	2.66	2.52	1.547
Nephelite	21, 23, 24, 25	23	0.079	2.6	2.81	1.540
Albite	27, 30, 32	30	0.134	2.62	2.69	1.530
Muscovite	29, 31, 32	31	0.143	2.83	3.07	1.574
Biotite	33, 33, 34, 36	34	0.171	2.90	3.11	1.605
Labradorite	40, 44, 45	43	0.269	2.71	2.90	1.563
Tremolite	42, 44, 44	43	0.269	3.0	2.90	1.601
Spodumene	44, 45, 46, 46	45	0.293	3.1	3.11	1.667
Tourmaline	45, 46, 48, 48	47	0.318	3.25	3.15	1.636
Anorthite	46, 48, 51	48	0.331	2.76	3.02	1.582
Augite	47, 48, 48	48	0.331	3.3	3.20	1.708
Rhodonite	48, 49, 49, 51	49	0.344	3.6	3.70	1.731
Olivine	49, 49, 49, 51	50	0.357	3.3	3.20	1.670
Topaz	47, 49, 51, 52	50	0.357	3.5	3.36	1.633
Cyanite	50, 50, 51, 53	51	0.371	3.6	3.65	1.723
Titanite	50, 52, 52, 53	52	0.384	3.45	3.24	1.947
Epidote	51, 53, 54, 54	53	0.398	3.45	3.24	1.750
Almandite	53, 54, 54, 56	54	0.412	4.25	4.20	1.790
Zircon	53, 56, 58, 58	56	0.441	4.7	2.73	1.958

[a] A. F. Rogers: Study of Minerals and Rocks. New York, 1921. McGraw-Hill Book Co.

The apparatus[18] employed to obtain contact-angle measurements was of the type designed by Taggart, but altered to accommodate the drop method of taking angles instead of the captive-bubble method. A drop of liquid was placed on a flat, dry mineral surface, shaken to equilibrium, photographed with 7 diameter magnification, and the angle read with a protractor from the photograph. Nearly 200 photographs of drops were taken by holding against the ground glass of the camera squares of sensitive bromide paper which were developed immediately The drops were of a solution made to simulate that used in the flotation cell and consisted of 0.5 gram of sodium oleate and 0.2 gram of terpineol per liter of water (pH 6.5).

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[18] Taggart, Taylor and Ince: Reference of footnote 6, 285.

The drop method of taking contact angles presents numerous small mechanical difficulties, which may be largely overcome with patience and practice. Table 2 shows individual angle readings, each reading being the average of the two angles of each drop. The term, $1 - \cos \theta$, is given for each mineral, and is a measure of the unwettability of the mineral.

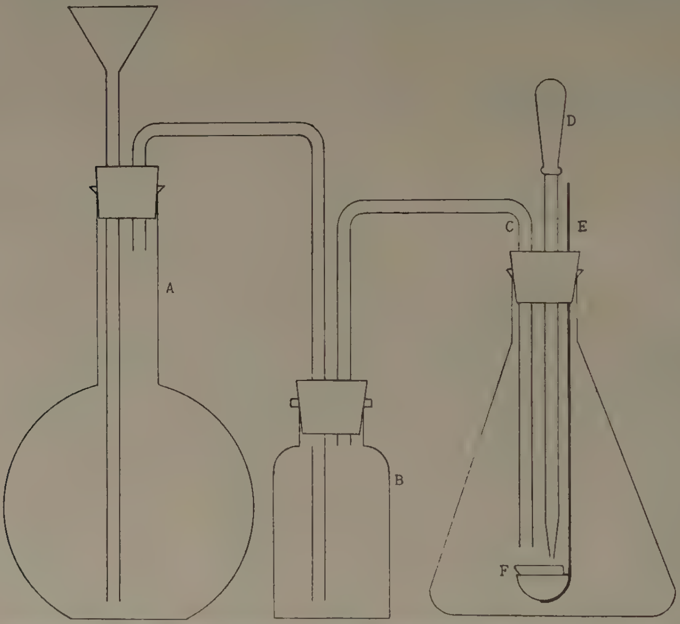

FIG. 3.—APPARATUS FOR OBSERVING CONTACT ANGLE OF DROP OF LIQUID ON MINERAL SURFACE IN ATMOSPHERE OF CARBON DIOXIDE.

A, carbon dioxide generator; *B*, calcium chloride container; *C*, carbon dioxide inlet; *D*, dropper; *E*, brass spoon; *F*, mineral.

Since the criticism was made that mineral surfaces might be contaminated by dirt and grease in the air, a drop of water was observed on zircon in an atmosphere of CO_2 gas (Fig. 3). A zircon crystal was mounted with sealing wax in a brass dipping spoon. The spoon with the mounted crystal was rapidly washed in hot NaOH solution and then in hot running tap water in order to remove any grease that might be present. A rough unglazed porcelain insulator was washed in hot NaOH solution, and then in scalding hot running tap water. A flat surface of the mounted zircon was then ground on the porcelain insulator under warm running tap water until the surface appeared frosted.

Rubber gloves were worn, but the mineral surface was at no time touched by the gloves. After being cooled in running tap water, the mineral, while wet, was placed in a 500-c.c. Erlenmeyer flask, directly under a stream of CO_2 gas, which had been flowing into the bottom of the flask for 45 min. The mineral was manipulated entirely by the handle of the spoon, which was mounted vertically in the loosely placed rubber stopper of the flask, together with a long glass dropper, and the CO_2 inlet. The crystal surface was placed directly in the stream of gas and permitted to dry. When dry, a drop of tap water was caused to fall from the dropper to the mineral surface.

The CO_2 gas had been generated with chemically pure hydrochloric acid, marble chips and tap water in a liter flask, and passed through ¼ lb. of C.P. calcium chloride into the Erlenmeyer flask.

The entire experiment was repeated three times, and certain facts noted. As the liquid dried from the mineral there was no tendency for the liquid to heap itself into drops. The film became so thin that it appeared only as a slight polish on the frosted mineral surface, and then it disappeared altogether. When the drop of water fell on to the dry surface, there was little tendency to spread, and an appreciable contact angle resulted.

Another type of contact-angle experiment was conducted with oil under water. Drops of 30° Bé lubricating oil were placed on freshly ground surfaces of microcline, sp.odumene, amphibole, topaz, zircon and cassiterite. In every case the oil gradually spread to a low contact angle. No difference between angles could be distinguished.

The minerals were then placed in water. The oil on the microcline drew up into a sphere which could be easily shaken off the mineral. On the spodumene, the oil contracted until an appreciable oil-solid contact angle was apparent. The oil on the cassiterite showed no tendency to contract. On the zircon, the oil showed no contraction, but seemed to continue to spread under water. Topaz may have showed a slight tendency to contract, but when the water was made acid with HCl, the oil contracted noticeably.

The contraction of the oil undoubtedly was due to a greater attraction of the mineral for the water than for the oil. The water crowded the oil from the mineral surface on the more wettable silicates, but not on the less wettable, such as zircon. The results of this experiment verify the results obtained with liquid drops on dry solid surfaces.

Correlation of Wettability with Density

The data in Table 2 were used to plot the curve in Fig. 4, in which a definite relation between density and wettability is shown. Low wettability, or from the standpoint of flotation, high unwettability, probably results from a high attraction between the atoms within the mineral, and from low attraction for the liquid at the mineral surface. The energy of the solid is probably consumed in the cohesive work of interatomic attraction, and little remains for the adhesive work of wetting. The same attraction that produces a high value for $1 - \cos \theta$ probably causes the mineral to possess a high density.

It was believed that as the attraction between the atoms increased, the distance from the center of one atom to that of an adjoining atom should decrease. The total volume allotted to each atom should, therefore, decrease, and the reciprocal of the volume, or the average density of the material occupying the space allotted to each atom should, therefore, increase. The writer attempted to calculate the density of the silicates by determining the density that should be given to each atom. This was done by dividing the density of each element by its atomic weight. Since the SiO_x is an acid radicle with the bond between its atoms different from that between it and the metal atoms with which it combines,[19] it was given an arbitrary fixed value. Calcium in the pyroxenes and titanite was arbitrarily given twice its value. The densities of the metal atoms and SiO_x radicles in each of the silicates were added, and the total divided by the number of atoms and radicles so as to obtain an average. This average was then multiplied by an arbitrary constant, 42, and a calculated density obtained. A comparison between calculated and actual densities is shown in Table 2. The writer realizes that these calculations are open to criticism, because of certain constants that were derived empirically and without explanation.[20] They do seem

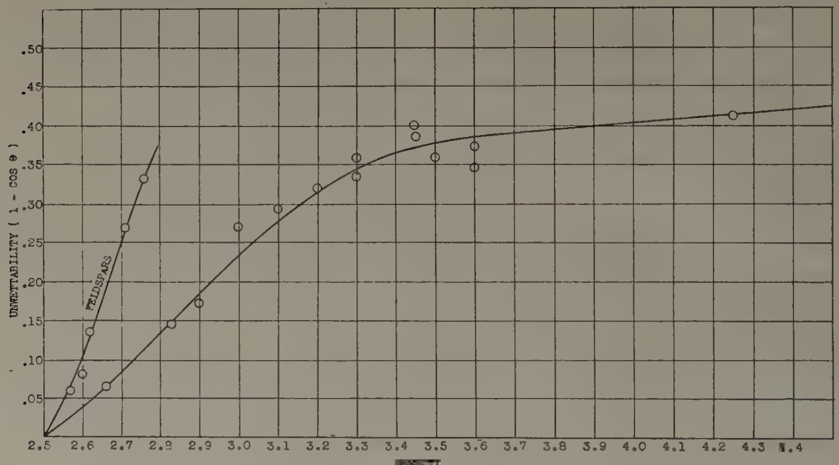

FIG. 4.—RELATION OF DENSITY TO WETTABILITY.

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[19] W. L. Bragg: *Ztsch. f. Krist.* (1930) **74**, 237–305.

[20] E. T. Wherry: *Amer. Min.* (1932) **8**, No. 1.

to show, however, that the density of a silicate mineral is based on interatomic attraction. The fact that the value of the wettability term, $1 - \cos \theta$, varies with density indicates that it also varies with interatomic attraction.

It has already been pointed out that the wetting of a solid by a liquid is governed by the relationship between the cohesive interatomic or intermolecular attraction within the liquid, and the adhesive attraction of the liquid for the solid. The attraction between solid and liquid depends as much upon the solid as upon the liquid; it is based on chemical affinity, portrayed by a tendency of the solid to combine with or dissolve in the liquid. A solid does not flow, but it partially controls the flow of liquid on its surface through its attraction for the liquid. And the energy of attraction of the solid for the liquid is that which is not held as interatomic attraction within the solid. In discussing solubility, where the energy of attraction is in terms of electrons, and solubility in terms of polarity, Hildebrand states:[21]

> If we substitute for the sodium [in sodium chloride] a metal like silver which parts less readily with an electron, we have in silver chloride a salt which is distinctly less polar. . . . The silicates of the heavier metals would be but slightly polar. . . . It seems further that the atomic volumes have a great deal to do with polarity. Where these are great, there would doubtless be more opportunity for the separation of the charges which produce polarity, the molecules being less compact, and we find, as a matter of fact, that the alkali metals, having the highest atomic volumes of all the elements, give highly polar compounds, followed in this respect by the halogens and the alkaline earths. The element giving the smallest atomic volume of all, carbon, gives the least polar of all compounds.

If a substance were composed entirely of elements of small atomic volume, one could reasonably expect the specific volume of the substance to be small, or the density high; and from this would follow low solubility and low wettability.

Correlation of Wettability, Density and Melting Point

Laurence says:[22] "The action of heat and of a solvent are not dissimilar in so far as they both loosen the directive forces holding the molecules in their normal crystal lattice."

The writer conducted an experiment which appears to illustrate this statement. Pieces of sheet copper, nickel, platinum and iron were heated to a bright red and allowed to cool until drops of distilled water placed on the metal surfaces would no longer boil. The contact angles of the drops on the metal were all very low; that of water on copper was approximately 0°, while that on platinum that previously had not been used for other purposes was greater than 0°. The angle of water on platinum appeared lower than that on iron, but higher than that on nickel. After five minutes of cooling, the contact angles were observed again, and their sizes roughly estimated and compared with one another. That of water on copper was now about the size of the initial angle on platinum, that on nickel was something like 15°, that on platinum was about twice that on nickel, and the angle of water on iron about three times that on nickel. It is probable that heating activates the metal, and thereby increases its wettability.

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[21] J. H. Hildebrand: Solubility, 96–98. New York, 1924. Chem. Cat. Co.

[22] A. S. C. Laurence: *Trans.* Faraday Soc. (1933) **29,** 1008.

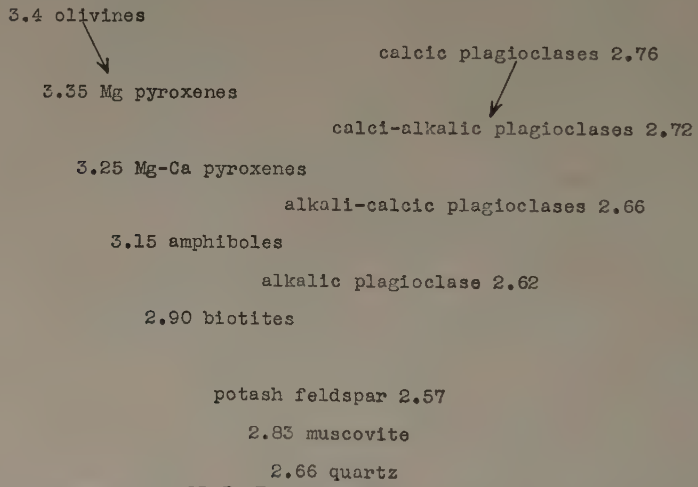

FIG. 5.—DIAGRAM AFTER N. L. BOWEN SHOWING ORDER OF CRYSTALLIZATION FROM MELT.

Figures designating average specific gravity were added to show relation of density to temperature of formation.

Hildebrand says:[23] "A solid having a higher melting point is less soluble at a given temperature than one having a lower melting point, provided the heats of fusion are not notably different in the two cases." Sidgwick[24] gives "Carnelley's rule, that of similar substances (that is where the partial pressure curves are not very different) the one which has the lower melting point is the more soluble."

These statements would lead one to expect the solubility of the silicates to vary inversely as the melting point. The wettability data obtained by reading contact angles substantiate this supposition. There are few definite figures on the solubility of silicates; that of quartz is given as 0.0160 grams per 100 c.c. of water,[25] while pseudo-wollastonite

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[23] Reference of footnote 21.

[24] N. V. Sidgwick: The Electronic Theory of Valency, 141. Oxford, 1929. University Press.

[25] A. Seidell: Solubilities of Inorganic and Organic Compounds. New York, 1919. D. Van Nostrand Co.

($CaSiO_3$) is but 0.0095 gram per 100 c.c. of water.[26] This is in line with what would be expected. Heavy silicates and oxides frequently are concentrated in placer and beach sands and are known to be very resistant to weathering.

From the data on the relationship existing between solubility, wettability and density, a correlation between density and melting point might be anticipated.[27] The addition of figures for density in terms of specific gravity to a diagram after N. L. Bowen[28] (Fig. 5) illustrating the olivines-pyroxenes-amphiboles-biotites discontinuous reaction series and the plagioclase continuous reaction series, shows this expected density-temperature relationship. An arrangement of the minerals according to wettability in Fig. 6 shows by comparison with Fig. 5 the correlation between temperature of formation and wettability.

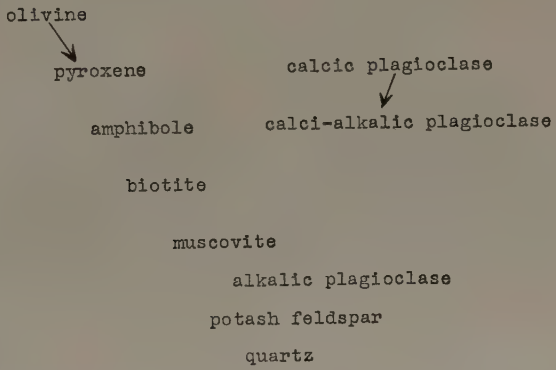

Fig. 6.—Diagram similar to Fig. 5 but arranged in order of wettability.

Effect of Collecting Agent

This paper has been devoted principally to a discussion of the relation of density to wettability, following the mention of a correlation between density and floatability. The influence of the collecting agent on floatability shown by both contact-angle determinations and flotation tests will now be considered.

Although it was known that the liquid used for contact-angle measurements would have its surface tension lowered to about 45 dynes per centimeter by the presence of the added oleic acid and terpineol, and that the liquid would therefore tend to have higher wetting power than distilled water, it was also believed that the effect of the collecting agent would be to increase the contact angle as in the captive-bubble test. However, it was found that the collecting agent did not increase, but actually seemed to decrease the contact angle. The amount of this decrease was 0° for quartz, 0° for microcline, 5° for spodumene, 7° for epidote, 7° for zircon, and 15° for black tourmaline.

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[26] C. H. Hodgman: Handbook of Chemistry and Physics. Cleveland, 1932. Chemical Rubber Publishing Co.

[27] E. K. Rideal: Surface Chemistry, 123–127. Cambridge, 1926. University Press.

[28] N. L. Bowen: Evolution of Igneous Rocks, 60. Princeton, 1928. University Press.

It was further observed that high floatability seems to be denoted either by a high contact angle with pure water, or by a large difference between an angle obtained with pure water and one obtained with soap solution. A high angle with pure water indicates low wettability; a high difference in angle seems to indicate high attraction between the collecting agent in solution and the mineral. In no case, however, was the contact angle with oleic acid solution less than 75 per cent of that with distilled water; and this permits an estimation of the relative importance of the collecting agent and of the natural wettability of the mineral. Fig. 7 shows a photograph of a drop of distilled water and a drop of oleic acid solution on epidote, the higher angle being that of the distilled water.

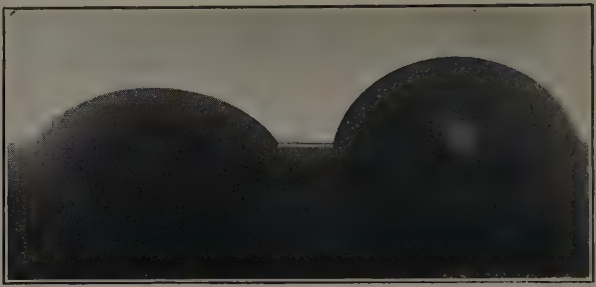

FIG. 7.—DROP OF OLEIC ACID SOLUTION (LEFT) AND OF DISTILLED WATER (RIGHT) ON EPIDOTE. × 7.

Difference between contact angles of drops is larger than usual with epidote, because of difficulty of bringing both drops to equilibrium at one time.

Difference between contact angles, as well as angle with distilled water, should be considered in estimating floatability.

The attraction between the solution containing a collecting agent and the mineral surface was taken to be the result of a tendency for the collecting agent to form an insoluble compound with atoms in the mineral surface.

The influence of oleic acid on floatability was thought by Gaudin[29] to result from a tendency of the oleic acid to form insoluble base-metal soap of low wettability on mineral surfaces possessing base-metal atoms. He suggested that the principal factor governing silicate floatability might be the ratio of the total number of valences of the atoms that would form insoluble soap to the total number of valences of those atoms that would not. The former may be considered those of the base metals, and the latter those of the alkali metals, water and silicon. Each silicon atom is considered as representative of a silica group to which all of the oxygen is allocated. (See Table 3.) This method of calculating valences is given in preference to Gaudin's "oxide" method, because it is in accordance with the modern conception of crystal structure as determined by X-ray analysis. Comparative values obtained by either method do not differ greatly.

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[29] A. M. Gaudin: Flotation, 343–386. New York, 1932. McGraw-Hill Book Co.

TABLE 3.—*Comparative and Calculated Floatabilities*

Order of Floatability (Actual)	Mineral	Formula[a]	Ratio of Floatable to Unfloatable Valences	Specific[b] Gravity (d)	Calculated floatability $d\sqrt[3]{\text{valence ratio}}$
1	Quartz	SiO_2	0/4	2.66	(0)
1	Orthoclase	$(Na, K)AlSi_3O_8$	3/13	2.57	1.57
2	Albite	$NaAlSi_3O_8$	3/13	2.62	1.61
3	Nephelite	$NaAlSiO_4$	3/5	2.6	2.19
4	Muscovite	$KH_2Al_3(SiO_4)_3$	9/15	2.83	2.39
4	Labradorite	$(NaAlSi_3O_8)_2$ $(CaAl_2Si_2O_8)_3$	0.69	2.71	2.41
5	Biotite	$KH_2(Mg, Fe)_3Al$ $(SiO_4)_3$	9/15	2.90	2.44
6	Spodumene	$LiAl(SiO_3)_2$	3/9	3.1	2.15
7	Anorthite	$CaAl_2(SiO_4)_2$	8/8	2.76	2.76
8	Augite	$CaMg(SiO_3)_2$ chiefly	4/8	3.3	2.62
9	Rhodonite	$Mn_4Ca(SiO_3)_5$	10/20	3.6	2.86
10	Titanite	$Ca(TiSi)O_5$	2/4	3.45	2.74
11	Tremolite	$H_2Ca_2Mg_5(SiO_3)_5$ with some $CaCO_3$	14/34 2/–	3.0	(3.19)
2	Epidote	$Ca_2(Al, Fe)_3OH$ $(SiO_4)_3$	13/13	3.45	3.45
13	Cyanite	Al_2SiO_5	6/4	3.6	4.12
14	Olivine	$(Mg, Fe)_2SiO_4$	4/4	3.3	3.3
15	Topaz	$Al_2F_2SiO_4$	6/6	3.5	3.5
16	Almandite	$Fe_3Al_2(SiO_4)_3$	12/12	4.25	4.25
17	Tourmaline (black)	$R_9Al_3B_2(OH)_2$ Si_4O_{19} R = Metal	24/18	3.25	3.58
17	Zircon	$ZrSiO_4$	4/4	4.7	4.7

The relation to floatability of the ratio of "floatable" to "unfloatable" valences (Table 3) is shown in Fig. 8. The valence ratio is apparently an index of the influence which the collecting agent exerts through the tendency of its polar group to combine chemically with the mineral.

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[a] Dana and Ford: Dana's Textbook of Mineralogy. New York, 1932. John Wiley and Sons.

[b] A. F. Rogers: Study of Minerals and Rocks. New York, 1921. McGraw-Hill Book Co.

However, this is only one of the factors influencing silicate floatability, the more important factor being the natural wettability of the mineral, as measured by density (Fig. 9).

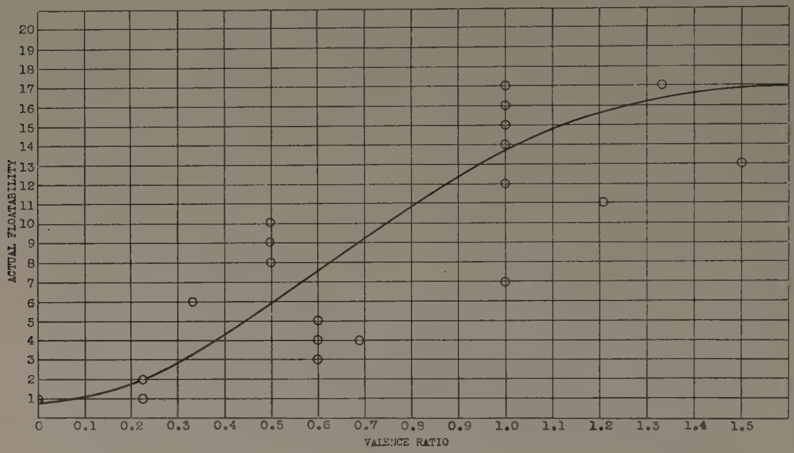

FIG. 8.—RELATION OF VALENCE RATIO TO ACTUAL FLOATABILITY.

If there were strong adhesion between water and a mineral, a condition which indicates solubility of the mineral in the water, there would no longer exist truly separate water and solid surfaces. However, if a mineral were but slightly wettable, the water and solid surfaces would

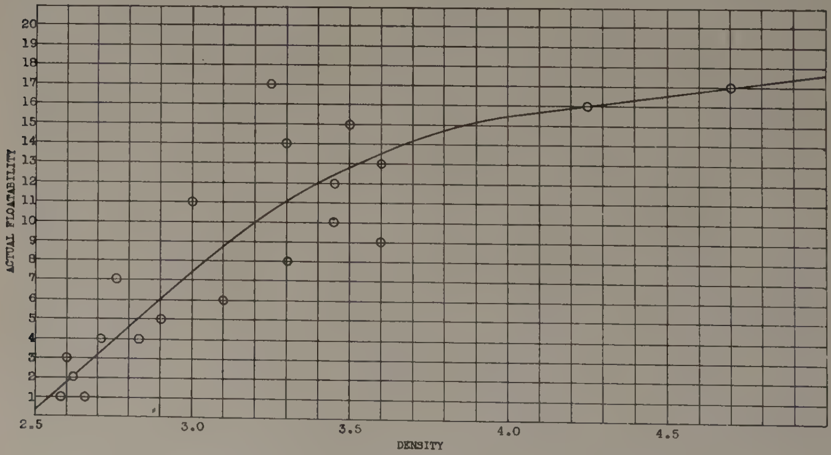

FIG. 9.—RELATION OF DENSITY TO ACTUAL FLOATABILITY.

still partially exist. The water surface against the mineral would then be somewhat similar to one against air in that it would offer a surface for the concentration of polar-nonpolar hydrocarbons such as oleic acid.

The oleic acid molecules would be expected to orient themselves in a monomolecular layer with their nonpolar groups toward the mineral, and their polar groups toward the water. Thus, the less wettable the mineral, the greater would be the concentration or adsorption of oleic acid at the mineral-water interface.

In the flotation of silicates, the oleic acid probably concentrates at the mineral surface, because of (1) the formation of insoluble metal oleates which depend on chemical reactions involving the polar group of the oleic acid molecule, and (2) the concentration or adsorption of the nonpolar groups of the oleic acid at the mineral surface because of the mutual insolubility of the nonpolar hydrocarbon and the mineral.[30] The high floatability of unusually insoluble minerals such as cassiterite must be attributed to the latter cause.

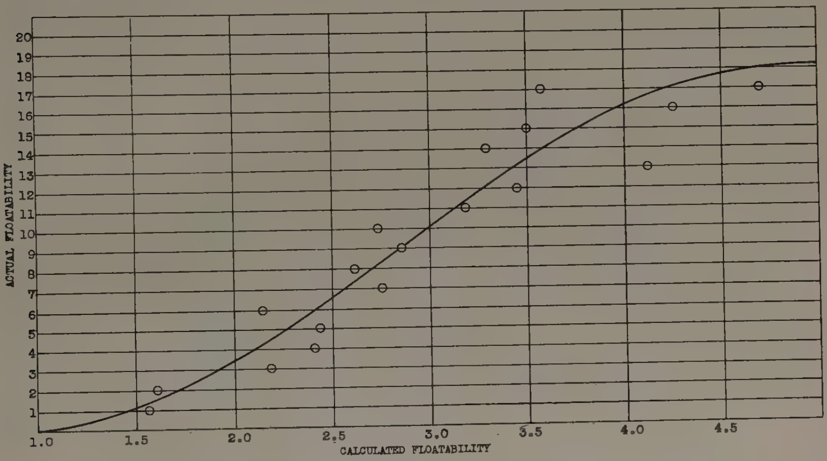

FIG. 10.—RELATION OF CALCULATED TO ACTUAL FLOATABILITY.

The comparative wettability of silicates is conveniently measured by their density, and their tendency to form insoluble soaps by their valence ratio. Since it was desired to find a means of predicting the floatability of silicate minerals by using the two factors, density and valence ratio, it was necessary to give each its proper weight, and the following empirical expression was formulated:

$$\text{Floatability} = \text{specific gravity} \sqrt[3]{\text{Valence ratio}}$$

Points indicating the relation of calculated to actual floatability (Fig. 10) appear to follow the curve $y = a(1 - \cos \alpha)$, where $\alpha = 180°$ when calculated floatability = 5.

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[30] Reference of footnote 21.

It should be remembered that differences in floatability are not entirely dependent upon the addition of flotation reagents or other types of conditioning, but exist from the time that the mineral crystallizes, as is shown by Bowen's photograph[31] (Fig. 11) of crystals in a melt turned to glass. Clusters of forsterite crystals are shown collected about three bubbles near the middle of the right-hand edge of the melt from which they had formed.

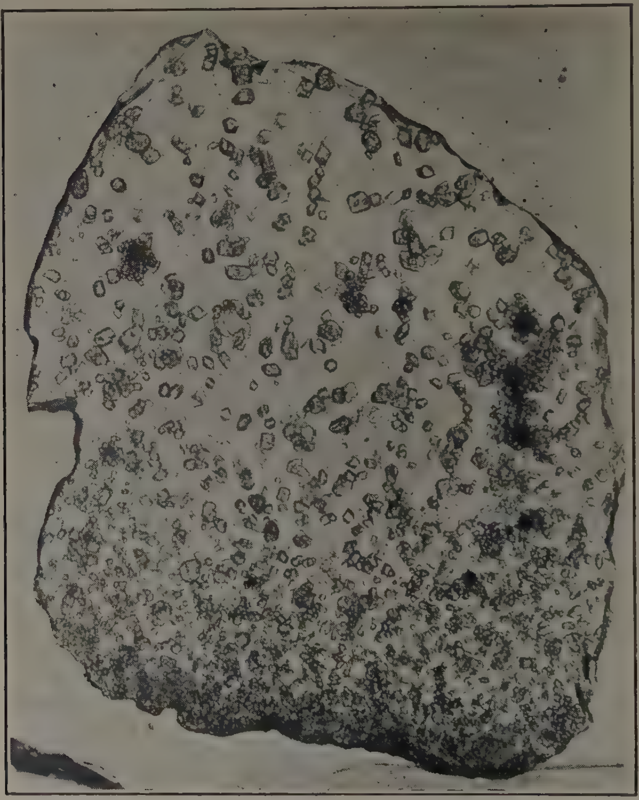

FIG. 11.—FORSTERITE CRYSTALS BUOYED BY BUBBLES IN MELT TURNED TO GLASS. (*Photograph by N. L. Bowen.*)

SUMMARY

1. The relative floatability of 20 silicates representing a wide variety of common types was determined by laboratory flotation tests.

2. Contact-angle readings were taken in order to determine the natural wettability of each silicate. The writer's conception of the wettability formula $Wc = \gamma_L (1 - \cos \theta)$ was given. A relation between unwettability $(1 - \cos \theta)$ and density was determined.

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[31] N. L. Bowen: *Amer. Jnl. Sci.* (1915) **39,** 179.

3. The action of the collecting agent on the contact angle was observed, and its role in flotation discussed.
4. Gaudin's valence ratio was combined with density in a formula to predict approximate floatability.

ACKNOWLEDGMENTS

The writer wishes to express his indebtedness to Dr. V. L. Ayres, Assistant Professor of Mineralogy at the Michigan College of Mining and Technology, under whose general direction much of the work was conducted, to Prof. C. M. Carson for providing laboratory accommodation in the Chemistry Department, and to Mr. R. F. Makens, of the Chemistry Department, for his interest and criticism.

The writer also wishes to express his appreciation of the assistance received from the faculty of the University of Wisconsin, particularly that received from Prof. R. S. McCaffery and Asst. Prof. E. R. Shorey, of the Department of Mining, who kindly furnished the use of laboratories and equipment, and also that received from Prof. Louis Kahlenberg, of the Department of Chemistry, who inspired a number of the ideas expressed in this paper.

DISCUSSION

(Arthur F. Taggart presiding)

A. F. TAGGART,* New York, N.Y.—We have done some work on the subject matter of Mr. Patek's paper at Columbia since the manuscript came through, and while we are much interested in the relationships he shows between actual floatability and density, we are far from satisfied as to the adequacy and correctness of his contact-angle measurements, as also of the early contact-angle measurements of Sulman, to which he refers, for the reason that we find that air-drying of a mineral surface will invariably produce a contact angle thereon, and also that with respect to sulfides—we have not worked with silicates—the contact angle produced is substantially independent of the sulfide. This fact, if we refer back to the work of Wark and Cox (p. 189, this volume) on contact angles with various sulfides in the presence of various collectors, indicates that the same collector is active with the different sulfides. In other words, the cause of the contact angle is air contamination, consequently the contact angles thus developed are not in any way due to or characteristic of the minerals themselves. We therefore think, in the investigation of contact angles, a polished surface produced under running water, which develops characteristic differences with different flotation agents, is properly usable for investigating differences in behavior of different minerals in the presence of a given reagent, or of different reagents on the same mineral, notwithstanding that we recognize that a polished surface may not be equivalent to a natural surface.

W. H. COGHILL,† Rolla, Mo.—Mr. Taggart mentioned that we might not have a natural surface similar to the polished surface. Similarly, we might have a liquid that is not just the same as in the flotation machine where there is vigorous agitation in the presence of mineral. It has been hard for me to see how a significant test for angle of contact can be made unless the liquid is in the identical condition of the liquid in the flotation machine.

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* Professor of Ore Dressing, School of Mines, Columbia University.

† Metallurgist, U.S. Bureau of Mines.

E. THOMAS,* New York, N.Y.—Is it not possible that there is more than one factor in the contact angles? I was astounded to find in a journal article that there could be four mutually immiscible liquids. Is it not possible that there can be a difference of contact angles due to different factors, in which one substance had a surface corresponding, say, to one liquid and another had a surface corresponding to another liquid?

A. F. TAGGART.—I think that might be true. The point that I am making now is that in one photograph Mr. Patek had drops of two different liquids on, presumably, the same surface, and he argues a conclusion as to relative floatability from the differences in contact angles against that one surface. I think that the contact angle has great significance in flotation, but I do not think that Mr. Patek's method of putting a drop on a particle that has been dried in grease-contaminated air tells him anything about the true surface that he is attempting to investigate. It rather tells him the behavior of his liquids on a contaminated surface. When he uses different gangue minerals it is possible that he may have differences in contamination, but our experience with sulfide surfaces is to the contrary. When we have prepared different sulfide surfaces by our method, that is, ground them under water, then polished them under water and at the end of the treatment known that they were clean from the fact that water formed a continuous film over them, and then permitted them to dry in air we have always obtained a surface contaminated in the same way, as indicated by the fact that a drop of water placed thereon made the same contact angle, irrespective of the sulfide surface. It made no difference how we dried them, whether in a vacuum, in a stream of ordinary cold air from a fan, in a stream of hot air, by letting them stand in the open laboratory, or letting them stand under a bell jar. Since differences develop with different sulfides when clean surfaces are treated, we believe that the similarity of behavior of air-dried material is in no way significant of the material.

MEMBER.—Do you get the same contamination with particles of quartz and galena that were cleaned under the same condition?

A. F. TAGGART.—We have not tested quartz. We have tested only the sulfides. We would not expect really to get a similar contaminating effect on quartz because when we positively contaminate quartz and galena by smearing, we get materially different contact angles, and, when the surfaces are examined under the microscope, distinctly different appearances.

In a paper by Mr. del Giudice there are photographs[32] of galena and of quartz contaminated by smearing each in similar fashion with the thumb. These photograph show that on the quartz the grease thus applied to the surface tends to form little islands, indicating a lack of wettability of the quartz by the grease, while on the galena surface there is a substantially complete, although somewhat irregular, covering of the surface by the grease. You get similar behavior when you apply oil to the two surfaces under water. On quartz the oil stands up as a spherical droplet; on galena it spreads out into flattened spherical segment. This demonstrates a distinct difference in the wettability by oil and water of the two surfaces.

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* Patent Lawyer and Chemist.

[32] Microscopy in Flotation Research. See page 447, this volume.

A. Legsdin,* New York, N.Y.—How does the author explain the difference in flotation of feldspars and silicates? What is the difference in behavior between the feldspars in one curve and the silicates in the other?

J. M. Patek.—Differences in flotation between the two reaction series depend on two factors: (1) the liquid-solid surface tension and (2) the formation with oleic acid of an insoluble substance on the mineral surface.

A. Legsdin.—What is this difference due to? Is it of chemical nature or is it due to a difference in the structure? If we take the compositions of the feldspars and of the silicates, there is a similarity in the compositions.

J. M. Patek.—It is due to both the chemical and physical factors.

A. Legsdin.—The feldspars, for instance, represent combinations in composition similar to many other silicates, we have similar combinations so far as chemical factors are concerned. I do not see a reason for the difference in floatability.

J. M. Patek.—You are talking about all the feldspars.

A. Legsdin.—I think that is what you mean. Were not all the feldspars represented by the curve?

J. M. Patek.—That was wettability.

A. Legsdin.—To what factors is this difference due?

J. M. Patek.—Those curves were made with an oleic acid solution and the differences are both physical and chemical, more physical than chemical, as shown by the formula

$$\text{Floatability} = \text{specific gravity}\ \sqrt[3]{\text{valence ratio}}$$

and also by the fact that where drops of distilled water and oleic acid solution are placed on the same mineral, and the oleic acid enters into combination with the base metal in the mineral, the difference in contact angle between the two drops is not very great. There may be up to about one-quarter reduction in contact angle due to pulling out of the drop, because of chemical interchange.

A. Legsdin.—I still do not see why there should be much of a difference. When we consider the garnets or the pyroxenes or the feldspars, we have minerals of similar composition, we have aluminum and silicon, so why should there be a difference in the wettability? Of course, in some of the silicates we are going to have iron also, or some other elements. Just what causes the difference in the floatability of those two groups? Why should the feldspars be in a group by themselves and the rest of the silicates in another?

J. M. Patek.—Independent differences exist in the wettability of the two reaction series.

A. Legsdin.—Then to what difference is this wettability due?

J. M. Patek.—That takes into consideration factors that would have to be analyzed before the question could be answered.

C. E. Locke,† Cambridge, Mass.—I look upon Mr. Patek's work as distinctly pioneering. I think we all agree with Professor Taggart that contact angle is not the entire story when it comes to determining floatability, but is an excellent indication, at least one of the factors of what we may expect when we come to actual flotation of the minerals.

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† Professor of Mining and Ore Dressing, Massachusetts Institute of Technology.

I noticed that in the preparation of the minerals they were carefully washed with hydrochloric acid, I think, and then all the fine slime was removed by decantation. Is there any possibility that that hydrochloric acid would have an effect upon the surface, to make the surface different from the original surface? Also, would the removal of the fine slime have any effect? What was the strength of the acid solution used; also, the relative range of size of material treated? In other words, how fine did it go?

J. M. Patek.—The acid was six normal hydrochloric acid, and the sand was 100 per cent minus 80 mesh and 92 per cent plus 200 mesh in size.

J. M. Patek (written discussion).—The relative contact angles recorded in this paper were made solely to obtain a scale of relative wettability, but it is questionable whether absolute angles will ever be determined accurately by any method in which the angle is read directly. Perhaps eventually we will turn to the work of Harkins and others on the wetting of pigments and powders, and, as they are now doing, will measure the energies of adhesion and immersion of different solids in water, rather than the contact angles. The assumption that the captive-bubble test is suited to determine the contact angles of true mineral surfaces rather than merely to measure the degree of surface contamination has not assisted the study of natural surfaces. I believe that certain observations in connection with this test have been allotted unwarranted significance. The detection of a slight film of undetermined nature on a sulfide surface which has first been polished wet and then subjected to air is an example. In the flotation of sulfides the purely chemical factors are of such importance that the more physical need scarcely be considered. However, this is not the case with oxides.

Magnetic Concentration of Ores

By R. S. Dean,* Member A.I.M.E., and C. W. Davis†

Development of the Art of Magnetic Separation

The purification of iron ores by means of hand magnets dates back more than 100 years. The first record in the United States Patent Office of a machine for the magnetic separation of ores shows that on Feb. 20, 1849, U.S. Patent 6121 was issued to Ransom Cook for improvement in electromagnetic ore separators. The patent covered the use of a revolving cylinder, or drum, with poles of electromagnets in its periphery for the purpose of separating magnetic oxide of iron from substances with which it might be associated, by causing the electromagnets when revolving as component parts of the cylinder to be successively charged to take up the ore and discharged to part with it.

Since this patent was issued the science and technique of magnetism have advanced more than most other fields of endeavor. The development of electrical machinery has required and obtained better and better magnetic materials, and the science of applied magnetism is as completely and satisfactorily developed as any branch of physics. Nevertheless, the patent of Ransom Cook, issued in 1849, comes close to covering everything that is now being applied in magnetic separation. The cynic may conclude that this proves the uselessness of modern science and may point in confirmation to flotation, which has grown in a few years, practically without benefit of science, to be the most versatile and useful of concentration methods.

On more careful study, however, it appears that an accurate determination of the magnetic properties of minerals and their amenability to control by mechanical and heat treatments has not heretofore been made. The first essential, therefore, in the design of improved magnetic separators has been missing. In spite of this hiatus in scientific data inventors have been busy, and patents are on file covering mineral separations by means of nearly all known magneto-mechanical effects. Because of a lack of knowledge of mineral properties, most of these inventions never worked well enough to find commercial use. The Metallurgical Division

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of the Bureau of Mines has undertaken to fill this gap in the data on properties of mineral substances, and many interesting and important improvements in magnetic separation seem likely to be forthcoming. This new work on the magnetic properties of minerals will be discussed later. Meanwhile, it will be interesting to review briefly the present state of the art.

A convenient classification of magnetic separators according to magnetic phenomena may be made as follows:

1. Separation due to attraction of magnetic particles to a magnetic pole.
    - *a.* Attraction to a permanent magnet pole.
    - *b.* Attraction for the pole of a direct-current electromagnet.
    - *c.* Attraction for alternating-current electromagnet.
    - *d.* Attraction for an iron plate due to induced magnetism in the particles.
2. Separation due to movement of diamagnetic particles out of an intense magnetic field.
3. Separation due to movement of magnetic particles in a moving (usually multiphase) magnetic field.
4. Separation due to movement of charged particles in a magnetic field.
5. Separation due to reaction of conducting particles to a moving magnetic field.
6. Separation due to activity of magnetized particles in an alternating magnetic field.
7. Separation due to remanent magnetism.

A subclassification, according to mechanical features, may be made as follows:

I. Drum separators.
II. Belt and disk separators.
III. Reciprocating separators.
IV. Centrifugal separators.
V. Drag separators.
VI. Falling-stream separators.
VII. Other separators.

We cannot discuss each of the classes in detail but will refer briefly to each class and to the subclasses of 1-*b*, within which practically all the present commercial separators may be placed.

Class 1-*a*.—The class, using permanent magnets, is almost obsolete. U.S. Patent 104354, Nov. 15, 1870, to John Y. Smith, is particularly worthy of mention. It covered a belt of moving magnets with reversed poles in succession. The ore was dropped by the insertion of a keeper across the magnet poles.

Class 1-*b*-*I*.—This class, covering direct-current electromagnet drum separators, is the most important. The following named separators have especial significance either because of important features or considerable industrial application:

WENSTROM SEPARATOR.—U.S. Patent 373211, Nov. 15, 1887, to Jonas Wenstrom. This separator consists of an armature barrel revolving around a magnet, the barrel being composed of a series of longitudinal soft-iron bars alternating with nonmagnetic material.

BALL-NORTON SEPARATOR.—U.S. Patent 430058, June 10, 1890, to Clinton M. Ball and Sheldon Norton. In this separator the ore is thrown from the field in trajectories varying with the richness of the ore.

INDUCED-ROTOR SEPARATOR.—The earliest separator of this type appears to be that covered in U.S. Patent 901368, Oct. 20, 1908, to C. Q. Payne. The material is fed on a transversely laminated rotor placed between the poles of a magnet, which may be constructed to produce concentrated high-intensity fields.

Class 1-b-II—Belt and Disk Separators.—The forerunner of the widely used magnetic pulley was patented in 1870 by John Smith, and covered the use of a belt traveling around a magnet cylinder for the removal of magnetic material. The most important mineral separators in this group are based on the Conkling, first patented in 1889, which covers a feed belt with transverse belts operating in a magnetic field. Submerged belt separators, the performance of which will be taken up later, are of comparatively recent use. Separators in which horizontal rotating disks supplant transverse belts as a means of removing magnetic material were patented by John T. Dawes, U.S. Patent 780870, Jan. 24, 1905.

Class 1-b-III—Reciprocating Separators.—The Weatherby separator is representative of this class. It constitutes a reciprocating separating table and means for creating a magnetic field in the path of the material moving over the table.

Class 1-b-IV—Centrifugal Separators.—The Gröndal disk separator, U.S. Patent 812171, Feb. 6, 1906, to Gustaf Gröndal, belongs to this class. A rotating disk below the pole pieces of a magnet is fed by an upward stream of pulp. The magnetic material is attracted to the disk and thrown off by centrifugal force.

Class 1-b-V—Drag Separators.—The Davis magnetic log washer is a representative separator of this class. It is covered in U.S. Patent 1153087, Sept. 7, 1915, to Edward W. Davis. It is essentially an inclined trough through which the ferromagnetic material is conveyed upwardly. Magnet pole pieces extend continuously beneath the trough and conform to its curvature.

Class 1-b-VI—Falling-stream Separators.—Edison patented a number of falling-stream separators. The principle of all of them is the deflection of a stream of falling magnetic material by a magnet pole.

Class 1-b-VII—Other Separators.—The Wetherill separator for weakly magnetic materials should be mentioned here (U.S. Patent 708185, Sept. 2, 1902, to John Price Wetherill). In this separator the material is fed into a concentrated magnetic gap and the nonmagnetic material washed out with water. The magnetic particles are then removed by a scraper. The recently developed Granigg high-intensity separator is of sufficient novelty to be described later.

Class 1-c.—This class covers separators in which the attracting magnet is activated by an alternating current. As a result of the alternating field the particles are agitated and the gangue freed. This principle is employed in a number of separators. It was first covered in U.S. Patent 922800, May 25, 1909, to Walter B. Moore.

Class 1-d.—Two patents are on record in this class: U.S. Patent 403256, May 14, 1889, to H. H. Eames, and U.S. Patent 805289, Nov. 21, 1905, to H. E. Heath. It is doubtful whether either one is operable.

Class 2.—Separation due to the movement of diamagnetic particles out of an intense magnetic field. Elmer Gates has several patents on separators of this class, but they have never been successful commercially.

Class 3.—Separation due to movement of magnetic particles in a multiphase magnetic field. This method of separation, while never successful commercially, has been the subject of several patents which may be summarized as follows:

U.S. Patent 619636, Feb. 14, 1899, to Otto Alexander Paul Trüstedt covers a multiphase wandering field of force which carries away the magnetic particles in a direction different from that in which nonmagnetic particles move.

U.S. Patent 1066619, July 8, 1913, to Howard L. Isbell covers a multiphase field traveling longitudinally and a nonmagnetic cover having a longitudinal reciprocating movement and a transverse slope.

U.S. Patent 1074999, Oct. 7, 1913, to Oscar Alfred Zander covers a magnetic field traveling transversely to the direction of ore feed.

U.S. Patent 1463713, July 31, 1923, to William Morris Mordey is basically identical with U.S. Patent 1074999.

U.S. Patent 1564731, Dec. 8, 1925, to Joseph Wetherby covers passing the material to be separated between a multiphase magnetic field and an induced field.

U.S. Patent 1564732, Dec. 8, 1925, to Joseph Wetherby covers passing the material past two progressive multiphase fields of different strengths moving transversely to the flow of material.

U.S. Patent 1729589, Sept. 24, 1929, to William Morris Mordey covers details of treating material with a multiphase magnetic field.

Class 4.—Separation due to the movement of charged particles in a magnetic field has likewise not found commercial use but is covered in the following patents:

U.S. Patent 653344, July 10, 1900, to Elmer Gates covers charging the material and passing it through a magnetic field.

U.S. Patent 653346, July 10, 1900, to Elmer Gates covers passing a current through a moving body of mixture and diverting the conducting particles by a magnet.

U.S. Patent 794647, July 11, 1905, to Henry M. Sutton, Walter L. Steele and Edwin G. Steele covers a magnetically energized roller and a statically charged member to convey ore to the roller.

Class 5.—Separation due to the reaction of conducting particles to a moving magnetic field has been the subject of a number of patents as follows:

U.S. Patent 400317, March 26, 1889, to Thomas A. Edison covers separating conductors and nonconductors by causing them to fall through a magnetic field whose lines of force are constantly shifting whereby the trajectory of the conducting substances is shifted.

U.S. Patent 402684, May 7, 1889, to Hiram S. Maxim covers passing the material through a rapidly alternating magnetic field, whereby the conducting material moves out of the field.

U.S. Patent 411899, Oct. 1, 1889, to Richard R. Moffat covers an alternating-current magnet which attracts magnetic substances and repels nonmagnetic conductors.

U.S. Patent 731042, June 16, 1903, to Elmer Gates covers a rapidly moving magnetic field for repelling diamagnetic substances.

U.S. Patent 940282, Nov. 16, 1909, to George D. Rogers covers passing the material vertically through a polyphase coil and subjecting the mixture to a rotating magnetic field whereby the conducting particles are given a horizontal component.

U.S. Patent 1416634, May 16, 1922, to Willard C. Hall covers a magnetic pole and a movable conductive member whereby the metallic particles are attracted to the conductor.

U.S. Patent 1417189, May 23, 1922, to Joseph Bartholemew McCarthy covers a moving magnetic field causing metallic particles to rotate and separate themselves from other constituents.

U.S. Patent 1605117, Nov. 2, 1926, to Mitsuo Koizumi is similar to 940282.

U.S. Patent 1657405, Jan. 24, 1928, to Fred B. MacLaren covers passing metallic and nonmetallic particles through a polyphase field moving transversely across a gap.

U.S. Patent 1829565, Oct. 27, 1931, to Royal Lee covers passing a stream of ore vertically past a solenoid with horizontal axis, the solenoid being actuated with high-frequency current.

Classes 6 *and* 7.—Separation due to activity of magnetized particles in an alternating magnetic field and separation due to remanent magnetism. The only patent that might be interpreted as belonging to these classes is U.S. Patent 1256972, Feb. 19, 1918, to D. M. Barringer, which covers the movement of particles of magnetite out of an alternating magnetic field when separated from the magnet poles by a conducting plate.

The new H_c separators developed by R. S. Dean and C. W. Davis belong in these classes and will be discussed in detail later.

Magnetic Properties of Minerals

The data on magnetic properties of minerals available to the metallurgist have been meager and misleading. A table of tractive forces for a field strength approximately that of high-intensity magnetic separation (about 10,000 oersted) was published by W. R. Crane[1] in 1902 and is still the best guide in this field. At best such a table is of qualitative value only, since such important factors as packing effect and particle size were ignored, and it is incomplete in that it gives no indication of the variation of such tractive force with field strength and fails to include the other important magnetic properties. In order to establish a basis for expressing and using the results on magnetic properties of minerals now being made available by the investigations of the Bureau of Mines, it will be necessary to discuss a few elementary magnetic principles.

A magnetic field is most conveniently produced by passing a current through a helix, or solenoid, of wire. The strength of the magnetic field in the center of such a solenoid of mean cross-sectional area, a, is determined by the number of turns of wire per centimeter length n multiplied by the current c, in amperes flowing through the wire; in other words, by the ampere turns. The usual magnetic unit for magnetic field strength, written H, has until recently been called the gauss, and this name is still used very commonly although the name "oersted" was recommended in 1930 by an international commission on electrical and magnetic units. It is related to the ampere turns (nc) and dimensions of the coil as follows: For long slender coils $H = \frac{4\pi nc}{10a}$. As the ratio of length to diameter of the solenoid becomes less than about 12:1, the demagnetization effects require the addition of correction factors.

Such a solenoid acts like an ordinary magnet with poles at its ends; the polarity is determined by the direction in which the electric current circulates in the windings of the solenoid; reversing the direction of the electric current reverses the direction of magnetization. The strength of a solenoid magnet, that is, the amount of pull it exerts, is not noticeably affected when the space within the helix is filled with most substances (for example, such as wood, glass, brass, cloth, liquids), but there exists a small group of substances that modify the magnetic strength or pull when inserted within the helix of the solenoid. Substances that have this property are called "magnetic," and of these magnetic substances the majority increase the strength of the solenoid. The commonest and easiest specification of the degree to which a given substance is magnetic is to indicate by how much it multiplies the original field strength when it fills the space within the helix; that is,

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[1] *Trans.* A.I.M.E. (1902) **31,** 450-

$$B = \mu H$$

or the so-called "magnetic induction" B, the new strength of the filled solenoid which is now designated as "electromagnet," is μ times the original field strength H; μ is called the "magnetic permeability" of the substance. The magnetic induction is measured in terms of a unit called the gauss (which in size is the same as the oersted).

Faraday discovered that practically all substances have some slight magnetic effect, although this effect is so feeble in most cases that it would multiply the pull of the solenoid by less than about 1.0001 (or 0.9999 for so-called "diamagnetic" substances). The permeability, as already defined, is therefore employed only for characterizing a comparatively few highly magnetic substances (which for convenience may be called "ferromagnetic"), while the vast majority of other substances have their susceptibility to magnetism specified otherwise. Consider, for example, bornite, which, according to W. R. Crane's table has $\mu = 1.0021$ to 1.0027; if this sample of Crane's bornite powder were made the core of an air solenoid, say of 500 oersted field strength, we would have

$$B = \mu H = 1.0025 \times 500 = 501.25 \text{ gauss}$$

(if μ were independent of field strength, which is a questionable assumption even in this case). Clearly, bornite and similar substances would be of no interest as substitutes for soft iron in the core of an electromagnet. Hence it is customary to say that the field in such cases is the original field increased by the slight magnetization caused by the field; that is,

$$B = H + 4\pi I,$$

where I is the intensity of magnetization of the bornite powder and is directly related to, in fact derived from, the usual conception of the pole strength of a magnet:

$$I = \frac{ml}{v}$$

where m is the "strength of a magnetic pole," l is the distance between the north and south poles and v is the volume of the magnet. The pole strength has no other name, but when I is multiplied by 4π it gives the number of gauss, hence $4\pi I$ may be called the "magnetization density." Dividing by H the equation just given leads to:

$$\frac{B}{H} = 1 + 4\pi \cdot \frac{I}{H}$$

or

$$\mu = 1 + 4\pi K$$

which shows the relation between the permeability μ, previously defined, and the susceptibility K, which is the more suitable for all questions relating to magnetic separation, because K and not μ is involved in the mathematical formulation of the adherence of magnetic particles to strong magnets and of the forces acting on magnetic particles (such as grains of ore) in the field of a magnetic separator.

In textbooks on magnetism, it is usually stated that the energy stored in a magnetic field is $\frac{B^2}{8\pi}$, and from this it is deduced that the tractive power of a magnet is $\frac{B^2}{8\pi}$. Both these statements are correct if B is understood in the proper sense. To illustrate: Take a toroid (a ring of circular cross-section) of soft iron; magnetize the ring to saturation by passing a heavy current through windings on its upper half. The magnetic induction B circulating around the ring can be determined by simple means. Now cut the toroid in half, and it will be found that the upper half will hold the lower half with a pull almost but not quite equal to $\frac{B^2}{8\pi}$ (expressed in the proper units), provided the cut surfaces are flat and highly polished. In the uncut ring B would be, say, 20,000 gauss; in the ring with the minute air space between the polished surfaces at the cut, B would be 19,500 gauss; the weight necessary to pull the lower half from the upper half would be:

$$\frac{(19{,}500)^2}{25.1} = 15{,}149{,}000 \text{ dynes} = 33.9 \text{ lb. per sq. cm. of area of contact.}$$

If a sheet of writing paper were put between the surfaces of contact, B would decrease to about 18,000 gauss and the pull per square centimeter of contact area would be 12,908,000 dynes, or 29.0 lb.; if a ½-in. layer of paper or wood separated the two halves of the toroid, B would be as low as 4000 gauss, and the pull would be only 1.4 lb. per sq. cm. of cross-section. If the lower half of the toroid is removed entirely, there would be an external magnetic field due to the magnetic induction in the iron, and this field at the horizontal flat surfaces (the poles) of the half toroid would be equal to the induction in the iron B, which might vary from 1000 down according to the distance apart of the poles (with the magnetizing current always at its original value, of course). This half toroid with its winding is a strong electromagnet for which the field strength H at its poles is taken as having been determined as 1000 oersted; suppose its poles are dipped into Crane's bornite powder of $\mu = 1.0025$, how much will adhere to the poles of the electromagnet?

Substituting in the formula $\frac{B^2}{8\pi}$ or $\frac{B_1B_2}{8\pi}$ would give:

$$\frac{1000 \times (1.0025 \times 1000)}{8\pi} = 1{,}002{,}500 \text{ dynes,}$$

or 0.45 lb. per sq. cm. of pole area, but a trial would show at once that even so strong an electromagnet could not lift this amount of bornite powder. The error is in using the permeability $\mu = 1.0025$ instead of the susceptibility $K = \frac{0.0025}{4\pi} = 0.0002$, as may be seen by trying to lift sawdust, glass, or other substance of permeability $\mu = 1.000$ or susceptibility $K = 0.0000$; the use of μ in this case would give practically the same 0.45 lb. per sq. cm. of pole area already calculated, using $B_2 = 1000$ and $B_1 = 1000$, but would become 0 if $B_1 = 1000$ and $B_2 = 4\pi I = 0$. The rule then for substances that are not themselves magnets (or have no source of magnetomotive force like a current-carrying coil) is to use $B_2 = 4\pi I$; thus for bornite the formula would be

$$\frac{4\pi IH}{8\pi} = \frac{IH}{2} = \frac{KH^2}{2}$$

(where H is now written in place of B as the field strength caused by the electromagnet), which would give:

$$\frac{0.0025}{12.57} \times \frac{(1000)^2}{2} = 99 \text{ dynes},$$

or 0.000044 lb., or about 19 mg. per sq. cm. pole surface, a pull that is not sufficient to sustain the weight of a particle of bornite.

Similarly, a mineral particle in a magnetic field, say, of a direct-current magnetic separator becomes magnetized to a value $4\pi I$ and is attracted to stronger parts of the field, that is, to the poles of the electromagnet producing the field, by a force

$$\frac{d}{dx}\frac{4\pi IH}{8\pi} = \frac{d}{dx}\left(\frac{KH^2}{2}\right) = KH\frac{dH}{dx},$$

where H and $\frac{dH}{dx}$ are respectively the strength and gradient at the part of the field occupied by the particle. The dynamic action of this force, in other words the motion produced by this force, depends on a number of factors and cannot be calculated with accuracy. However, given a certain field, the formula shows that the two powders may be compared by the ratio of their susceptibilities $\frac{K_1}{K_2}$, since $H\frac{dH}{dx}$ is fixed by the configuration of the field. This comparison, again, is only approximately correct because of several factors among which are: (1) that a magnetized particle distorts the original magnetic field, and (2) that the shape of the particle must be taken into account; that is, there is a form factor (called "demagnetizing effect").

It is thus evident that, although theory as yet is not developed sufficiently for an exact mathematical formulation and calculation of magnetic separation phenomena, enough is known to give a good qualitative and an approximate quantitative theory; thus, the forces on the particle are roughly proportional to the susceptibility K, and the potential energy in unit volume of the field between the poles of a magnet and a magnetized particle is proportional to IH, where H is the strength of field and I is the intensity of magnetization induced in the particle.

As stated above, the attraction of a magnet pole for a mineral particle is proportional to the intensity of the attracting magnet pole H_a, and of

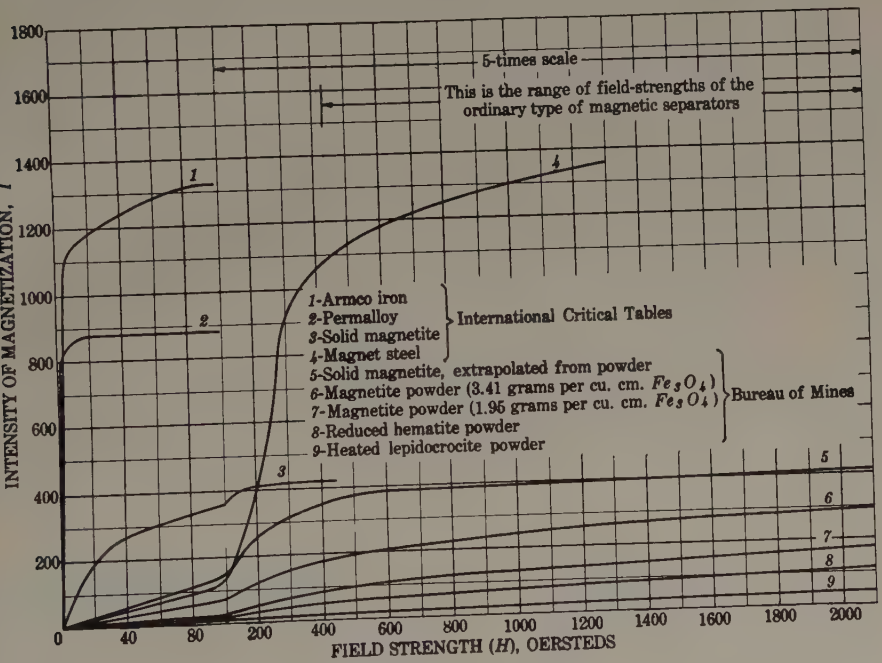

FIG. 1.—MAGNETIZATION CURVES.

the induced pole $H_i = 4\pi I$. The intensity of the induced magnetization for a given value of H_a is given by the magnetization curve. Fig. 1 shows such curves for a number of substances. The value of H for a given separation must be properly chosen; if, for example, we should want to separate a mixture of chatty sponge iron, made by reducing a low-grade ore, from unreduced magnetite, the magnetization curves for the two substances would appear about as shown in Fig. 2.

At H_2 the separation by tractive force would be impossible. At H_1 the sponge iron would be attracted away from the magnetite, but at H_3 the magnetite might be attracted from the iron if some means could be provided to prevent the attraction of both; centrifugal force, for example. If the magnetization curve were a straight line its slope would be the susceptibility K. This value, divided by density, is called the "mass" susceptibility $x = \frac{K}{d}$, and is the ratio of magnetic force to gravitational force acting on a particle. Hence, it is a measure of the extent to which a magnetic particle acted on by gravity will be affected by a magnetic attraction. It should be remembered, as Fig. 1 shows, that for strongly magnetic substances K is not a constant.

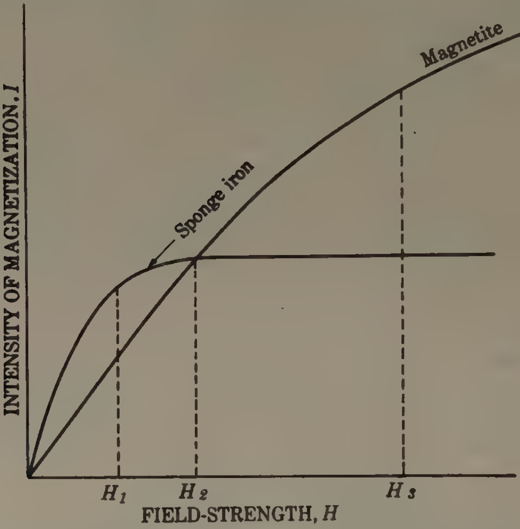

FIG. 2.—MAGNETIZATION CURVES, CHATTY SPONGE IRON AND MAGNETITE.

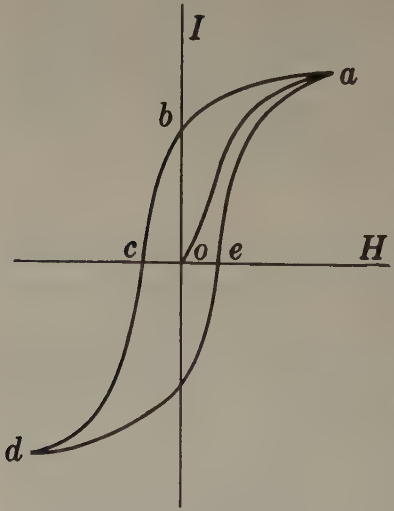

FIG. 3.—HYSTERESIS LOOP.

A thorough knowledge of the magnetization curves of minerals in the natural and treated states accordingly would enable many separations to be made which now are not deemed feasible.

If, after the value of H has been increased to a maximum, it is lowered, the value of I will not be the same as on the original application of the same field but will be somewhat more, as indicated by the curve AB (Fig. 3). When the field has been reduced to zero the value of I will be b. This is the intensity of residual magnetization I_r. If the field is reversed it will have to be increased, in the reverse direction, to c, before the specimen will act as though completely demagnetized. This value c is known as the coercive force H_c.

If, after the coercive force is reached, the field is further increased, the value of $-I$ will increase until saturation at d is attained. By again lowering and reversing H the original point a can be reached. Such a loop obtained by cyclically reversing the field is called a hysteresis loop. The values I_r and H_c are particularly significant for determining the properties of a magnetic substance. I_r indicates the intensity of magnetization shown by the specimen when the field is removed and H_c the strength of field necessary to demagnetize the specimen. It will be shown later that separation of minerals may be based on differences of these properties.

In order to describe completely the magnetic properties of a mineral specimen, therefore, we need to know the magnetization (or the normal induction) curve and the hysteresis loops at all field strengths. The values of I at some strength near saturation and I_r and H_c for a field strength near saturation will describe the properties of a specimen with sufficient accuracy for most, but not all, purposes.

The methods for the determination of hysteresis loops for minerals have been described in detail by Gottschalk and Davis.[1a] The values of I_{2000}, I_r, K_{2000} and H_c for several mineral specimens are given in Table 1

TABLE 1.—*Magnetic Constants for Mineral Powders*[a] *at a Field Strength H of* 2000 *Oersted*

Substance	Intensity of Magnetization, I_{2000}	Susceptibility, K_{2000}	Coercive Force, H_c	Remanence, I_r
Tool steel, solid	1,200	0.600	12	874
Tool steel, 60-mesh cuttings	214	0.011	12	20
Magnetite, crystal	560	0.280	2	4
Magnetite, 100 per cent, powder	222	0.111	6	56
Magnetite, pure, −325 mesh	199	0.100	49	24
Mineville magnetite	200	0.100	19	9
Mineville magnetite, grains oriented	220	0.110	19	13
Reduced hematite	72	0.036	400	34
Lodestone, California	92	0.046	269	39.1
Lodestone, titaniferous	137	0.069	142	39.0
Lepidocrocite, dehydrated	33	0.017	43	6.8
Lepidocrocite, reduced	100	0.050	88	10.3
Ilmenite, treated	4.8	0.0024	335	1.8
Pyrrhotite	4.6	0.0023	220	1.9

[a] Powders at a packing density of 2.5–2.8 grams per cubic centimeter and −60 + 250 mesh (except one designated as −325 mesh).

Tool steel, solid and as hack-saw cuttings, and pure crystallized magnetite have been included for comparison.

[1a] V. H. Gottschalk and C. W. Davis: An Apparatus for Determining the Magnetic Constants of Mineral Powders. U.S. Bur. Mines *Rept. of Investigations* (in prep.).

Pretreatment of Minerals for Magnetic Separation

All minerals receive some treatment before magnetic separation, even if only simple grinding. Even this grinding may have an important effect on the magnetic properties. It has been found, for example, that magnetite undergoes a definite change of magnetic properties with particle size. The smaller the particle size, the higher the coercive force and the lower the induction at any given field strength. Fortunately, a very simple relation has been deduced between particle size and coercive force. The coercive force is a linear function of the specific mineral interface. Fig. 4, taken from a recent paper by Gottschalk,[2] shows the relation of coercive force to specific interface of two pure magnetites (from the Ural Mountains and crystallized out of Hayden copper slags, respectively). Table 2 gives the complete magnetic data for Ural Mountain magnetite of several particle sizes.

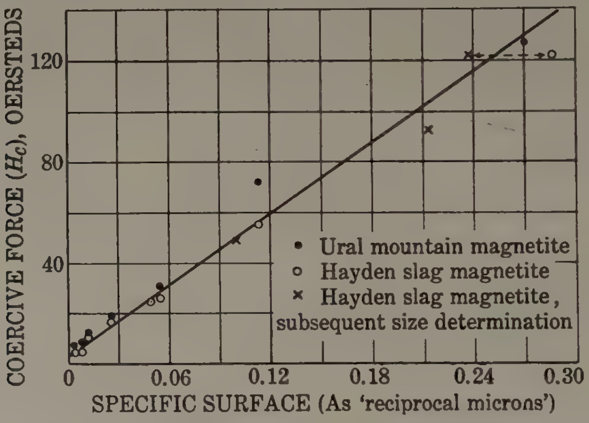

Fig. 4.—Magnetite powders—variation of coercive force with specific surface.

This dependence of magnetic properties on specific mineral interface has led to a general method for increasing the coercive force of minerals. Any magnetic material that can be produced below its temperature of substantial crystal growth will have a relatively high coercive force. Thus, if magnetite is produced by the reduction of hematite below 600° C. it has a coercive force of several hundred oersted. If the reduced hematite is heated to a higher temperature to bring about crystal growth the coercive force will fall, as shown graphically in Fig. 5. (Taken from the work of Donald Doan, Bureau of Mines Fellow at the Missouri School of Mines.)

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[2] V. H. Gottschalk: The Coercive Force of Magnetite Powders. U.S. Bur. Mines *Rept. of Investigations* (in prep.).

TABLE 2.—*Hysteresis Data for Ural Mountain Magnetite of Various Grain Sizes*

Field Strength H, Oersted	12.57 × Intensity of Magnetization, $4\pi I$, Gauss							
	1	2	3	4	5	6	7	
	−100 +150 Mesh	−150 +200 Mesh	−200 +270 Mesh	−270 +350 Mesh	−350 Mesh, Air-separated into Three Fractions			
2,700	2,849	2,789	2,743	2 619	2,825	2,503	1,691	2,174
1,745	2,742	2,678	2,626	2,496	2,682	2,349	1,580	2,033
1,260	2,598	2,526	2,475	2,343	2,504	2,177	1,471	1,889
870	2,358	2,284	2,234	2,112	2,246	1,952	1,337	1,710
500	1,850	1,786	1,748	1,661	1,760	1,561	1,115	1,414
246	1,232	1,194	1,177	1,146	1,222	1,151	880	1,111
136	804	789	789	794	864	878	721	912
77	523	523	532	558	626	695	614	778
48	362	371	384	419	489	592	555	703
34	279	293	309	348	422	542	525	666
19	180	197	215	259	333	475	487	615
4	81.2	110.1	126	171	247	418	450	559
−10	−22.2	−3.7	17.0	68.0	152.9	342	411	518
−41	−241.5	−226.0	−206.1	−155.8	−65.0	193.5	319	405
−97	−583	−555	−544	−500	−437	−144.0	113.7	147.0
−210	−1,068	−1,021	−1,002	−946	−934	−669	−283.6	−359.1
−440	−1,756	−1,693	−1,653	−1,554	−1,616	−1,353	−875	−1,107
−840	−2,319	−2,239	−2,187	−2,060	−2,180	−1,870	−1,259	−1,604
−1,230	−2,578	−2,501	−2,449	−2,314	−2,468	−2,139	−1,437	−1,841
−1,700	−2,734	−2,666	−2,614	−2,482	−2,665	−2,331	−1,564	−2,013
−2,700	−2,849	−2,789	−2,743	−2,619	−2,825	−2,503	−1,691	−2,174
$\frac{\text{Grams}}{\text{C.c.}}$ Fe_3O_4, packing density	2.71	2.67	2.63	2.52	2.74	2.47	1.71	2.28
Microns, mean grain size	180	134	92	40	18	9	3.5[a]	3.5[a]
H_c, oersted (by numerical interpolation)	6.2	9.6	12.4	19.4	31.7	73.1	126.5	127.0
B_r, gauss (by numerical interpolation)	51.6	77.6	94.5	144.4	220.1	397.3	438.9	547.3

[a] Remeasured subsequently to be of mean grain size 3.7 microns.

The hysteresis loops of ordinary 60-mesh magnetite, and of reduced hematite are shown in Fig. 6.

Methods of treatment based on this principle have been devised so that nearly any mineral containing iron can be treated to obtain high coercive force. Table 3 shows results of treating several minerals.

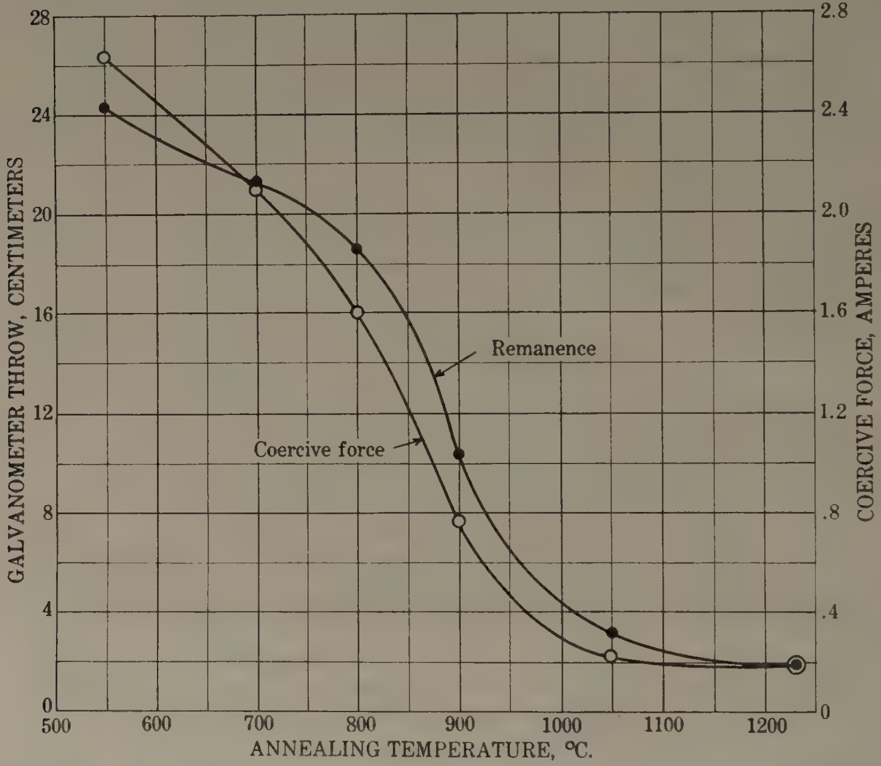

FIG. 5.—VARIATION OF REMANENCE AND COERCIVE FORCE FOR REDUCED HEMATITE (MISSOURI) AT VARIOUS ANNEALING TEMPERATURES.

To obtain high coercive force it is not necessary to convert the mineral completely into a highly coercive state, and as will be shown later it is possible to make separations of minerals with high coercive force and relatively low mass susceptibility. This circumstance indicates the possibility of flash roasting to convert hematite partially to magnetite or to gamma Fe_2O_3 at low cost. Preliminary experiments in this direction have given promising results. If, for example, hematite or limonite are passed rapidly through a tube at 1400° C., they attain a mass susceptibility of only about one-fifth that necessary for separation on the usual low-intensity separator (see p. 526), but the coercive force rises to 330 oersted, and the mass susceptibility is sufficient for separation on the H_c type of separators described later. It is impossible to estimate the cost of such treatment, but it would certainly be less than the conventional reducing roast.

Table 3.—*Effect of Heat Treatment on Magnetic Properties of Mineral Powders*

Mineral	Heat Treatment	H_c, Oersted	B_r, Gauss	Magnetization $4\pi I$	H Max., Oersted	Bulk Density
Magnetite.....	None	20	137	2,846	2,700	2.72
Magnetite.....	Heated in air	100	240	1,200	2,700	2.70
Hematite......	None	Too small to determine		Less than 12	2,000	
Hematite......	Reduced to Fe_3O_4 by heating in hydrogen	425	340	784	2,000	2.04
Lepidocrocite...	None	Too small to determine		Less than 12	2,700	
Lepidocrocite...	Heated in air to 370° C.	43	85	414	2,700	1.774
Lepidocrocite...	Reduced to Fe by heat in hydrogen	88	130	1,255	2,700	1.359
Ilmenite.......	None	Too small to determine		Less than 12	3,340	
Ilmenite.......	Heated in air	335	24.2	59.1	3,340	2.601
Pyrite.........	None	Too small to determine		Less than 12	2,700	
Pyrite.........	Light roast	600	80	180	2,700	1.5
Chalcopyrite...	None	Too small to determine		Less than 12	2,700	
Chalcopyrite...	Light roast	350	30	100	2,700	1.81
Arsenopyrite...	None	Too small to determine		Less than 12	2,700	
Arsenopyrite...	Light roast	300	85	230	2,700	3.11

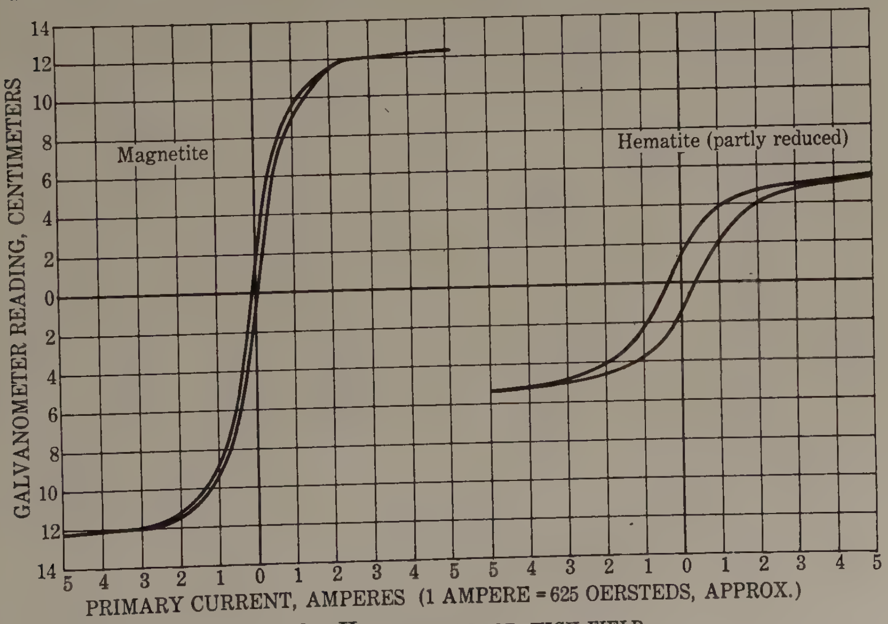

Fig. 6.—Hysteresis loop, high field.

Treatment to increase susceptibility has not yet been so well rationalized. It is well known, of course, that the reduction of iron oxides to the state of Fe_3O_4 produces markedly increased susceptibility. This treatment as applied to hematite is called "magnetic roasting" and has been discussed by Davis.[3] In general it consists in heating the hematite in a reducing atmosphere at 550° to 600° C. for about 20 min. The magnetic properties of the resulting material have been given in Figure 6 and Table 3.

The cost, including all operating charges, repairs and depreciation, but excluding the cost of magnetic separation, has been estimated at 83¢ per ton of concentrates for magnetic roasting of a hematite ore containing 40 per cent iron, so that a 63 per cent iron concentrate with a 95 per cent iron recovery may be produced.[4]

Low-temperature reduction of iron oxides to the metallic state has also been proposed as a step in the beneficiation of iron ores by magnetic separation for use in the blast furnace[5] or for use in direct iron and steel.[6]

In view of the expense of reducing roasts, it would be desirable to devise other means of increasing the susceptibility of hematite. The reaction between iron oxide, siderite and manganiferous spathic iron ore has been introduced recently in Germany for this purpose. It has been found that at 700° to 800° C. this reaction proceeds satisfactorily.

A line of attack more likely to yield results of general value would seem to be the conversion of ordinary ferric oxide (Fe_2O_3) into the cubic or gamma form, which has magnetic properties comparable to reduced

TABLE 4.—*Hysteresis Data for Dehydrated Lepidocrocite*

Field Strength H, Oersted	Magnetization $4\pi I$, Gauss[a]	Field Strength H, Oersted	Magnetization $4\pi I$, Gauss[a]
2,700	413	8	90
1,680	398	−5	74
1,175	377	−31	33
786	344	−82	−74
435	289	−177	−186
217	247	−392	−280
122	184	−750	−344
71	149	−1,172	−379
46	130	−1,640	−400
33	122	−2,700	−413
20	105		

Packing density 1.774 grams per cubic centimeter; H_c, 43 oersted; B_r, 85 gauss.

[a] $4\pi I = B - H$.

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[3] E. W. Davis: Univ. of Minnesota *Bull.* 9 (1921) 35–49.

[4] E. W. Davis: Concentration of the Mesabi Hematities. *Trans.* A.I.M.E. (1930) **90,** 362.

[5] W. H. Smith: Low-temperature Reduction of Ore. *Trans.* A.I.M.E. (1930) **91,** 419.

[6] C. W. Davis: Alternating-Current Magnetic Separation of Iron Ores. U.S. Bur. Mines *Rept. of Investigations* 3229 (1934) 37.

hematite. If the gamma hydrated iron oxide mineral lepidocrocite ($Fe_2O_3.H_2O$) is hydrated at 350° to 400° C., the resulting material has the magnetic properties given in Table 4.

In accordance with the general rule laid down for magnetic substances formed below their temperature of substantial crystal growth this gamma ferric oxide has a high coercive force Alpha hydrated ferric oxides do not give substances of so high a susceptibility on dehydration, but conditions usually can be found under which sufficient conversion to gamma hydrate takes place, so that dehydration will result in a high-coercive-force material of greater susceptibility than hematite.

It is probable that methods eventually will be found for cheaply and effectively converting the iron content of minerals into one of the highly susceptible forms.

So far we have not considered the feebly magnetic substances that may be separated by the so-called "high-intensity" separators. According to Frantz and Jarman[7] modern separators may employ fields as high as 15,000 oersted and will separate substances with a mass susceptibility as low as 10^{-5} c.g.s. units. This would correspond to a value for I of 0.75 gauss for substances with a density of 5. If the value of I at saturation is taken as 300 gauss for magnetite and the intensity of magnetization is proportional to the magnetite content, it is evident that 0.25 per cent magnetite in an inert material would make it amenable to separation in a high-intensity separator Most materials that are separated on high-intensity separators contain more iron than the equivalent of this magnetite content, and it is probable that they owe their susceptibility to small amounts of strongly magnetic material.

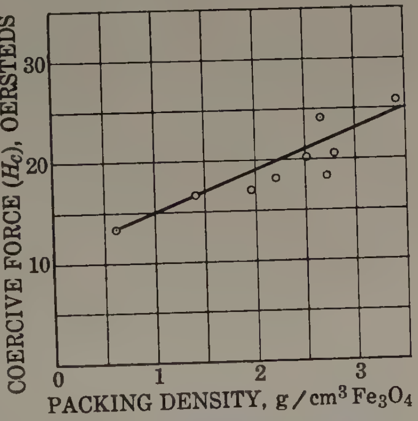

FIG. 7.—HAYDEN SLAG MAGNETITE VARIATION OF COERCIVE FORCE WITH PACKING DENSITY.

The question of the magnetic structure of feebly magnetic materials is of the greatest importance in determining the path for development in high-intensity separators. A dispersion of a strongly magnetic material like Fe_3O_4 in an inert nonmetallic matrix may behave as a magnetically soft material, or one with very low coercive force, and may have a comparatively low saturation value. Fig. 7 shows the effect of inert dilution on the coercive force of fine magnetite. Hematite, on the other hand, is apparently a homogeneous magnetic material of low susceptibility. No quantitative data are available, but it may easily be determined qualitatively that by magnetizing hematite at field strengths of the order of 10,000 oersted a coercive force of the order of 200 oersted is developed.

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[7] S. G. Frantz and G. W. Jarman: Magnetic Beneficiation of Nonmetallics. *Trans.* A.I.M.E. (1932) **102**, 122.

Field and Limitations of Existing Commercial Separators

Low-intensity Separators

Separators in this class are usually of either the drum, belt, or drag type. According to Davis (reference of footnote 3) materials to be amenable to treatment on a low-intensity separator must have a mass susceptibility of at least half that of magnetite. This limits their use at once to magnetite, scrap and sponge iron, roasted oxides and sulfides of iron.

The application of low-intensity separators to dry magnetite concentration is well illustrated in the operations of the Witherbee, Sherman & Co. mill at Mineville, N.Y., which has been described in detail by F. T. Myners.[8] Three types of separators are used: (1) a pulley type, which is used as a cobber to discard practically barren rock; (2) a drum type, which consists of a closed cylinder of brass revolving around stationary magnets covering two-thirds of the periphery of the shell (in which the magnets are so wound that adjacent poles are of opposite polarity, and the speed of the drum is said to be so adjusted that the time to pass from pole to pole is less than that necessary for particles of magnetite to lose their secondary magnetism, the result being a winnowing action which releases entrained gangue); and (3) a belt type, which is especially suited to finer materials.

Unfortunately no data are available on the actual field strengths used in any of these machines, but Bring[9] states that a field strength of 800 to 900 oersted is sufficient for the separation of coarse material, while 2000 oersted may be required to separate fine products. A middling from iron ore may be produced with a field strength of 400 to 500 oersted, and wet separation of crushed material may be effected at 800 to 900 oersted. The interpretation of the action of the reversed poles is probably wrong. Magnetite as a rule has a comparatively low remanent magnetism and, so far as we have been able to determine, drops to this low value instantaneously. The loss of the remanent magnetism itself is a matter of days, not fractions of a second. The stirring action is due, then, to the entire mass falling away from the drum and being reattracted by the next magnet. The remanent magnetism may be sufficient to make the particles turn end for end, but in the authors' opinion the speed regulation is a matter of centrifugal force not loss of remanent magnetism. This is

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[8] F. T. Myners: Magnetic Concentration Methods and Costs of Witherbee, Sherman & Co., Mineville, N. Y. U.S. Bur. Mines *Inf. Circ.* 6624.

[9] G. G. Bring: Investigations on Magnetic Separators. *Jernkontorets Ann.* (1932) 116, 255–278.

TABLE 5.—*Magnetic Concentration of Fine and Coarse Magnetite Concentrates from the Richard Mine, Wharton, N.J.*

Size, Mesh	Product	Fine Concentrates							Coarse Concentrates						
		Weight, Per Cent	Assay, Per Cent			Percentage of Total			Weight, Per Cent	Assay, Per Cent			Percentage of Total		
			Fe	SiO_2	P	Fe	SiO_2	P		Fe	SiO_2	P	Fe	SiO_2	P
– 8	Concentrates........	93.0	66.38	3.90	0.181	99.1	54.0	54.0	90.6	67.17	3.24	0.142	99.0	38.8	38.9
	Tailings............	7.0	7.35	44.14	2.043	0.9	46.0	46.0	9.4	6.51	49.36	2.14	1.0	61.2	61.1
	Composite.........	100.0	62.26	6.72	0.311	100.0	100.0	100.0	100.0	61.47	7.58	0.329	100.0	100.0	100.0
– 48	Concentrates........	87.1	70.61	0.79	0.052	98.8	10.3	14.5	86.2	70.51	0.56	0.028	98.9	6.8	7.3
	Tailings............	12.9	6.09	46.74	2.06	1.2	89.7	85.5	13.8	5.02	51.44	2.21	1.1	93.2	92.7
	Composite.........	100.0	62.26	6.72	0.311	100.0	100.0	100.0	100.0	61.47	7.58	0.329	100.0	100.0	100.0
–100	Concentrates........	86.3	71.20	0.43	0.039	98.7	5.5	10.9	85.1	71.39	0.28	0.02	98.8	3.2	5.2
	Tailings............	13.7	5.91	46.35	2.022	1.3	94.5	89.1	14.9	4.62	49.26	2.09	1.2	96.8	94.8
	Composite.........	100.0	62.26	6.72	0.311	100.0	100.0	100.0	100.0	61.47	7.58	0.329	100.0	100.0	100.0
–200	Concentrates........	85.9	71.49	0.35	0.031	98.6	4.5	8.7	85.0	71.49	0.28	0.018	98.8	3.2	4.6
	Tailings............	14.1	6.02	45.53	2.014	1.4	95.5	91.3	15.0	4.60	48.93	2.09	1.2	96.8	95.4
	Composite.........	100.0	62.26	6.72	0.311	100.0	100.0	100.0	100.0	61.47	7.58	0.329	100.0	100.0	100.0

mentioned as an example of the need for more complete magnetic data on minerals as an essential element in the design of separators.

The capacities of the three kinds of machines described are given as follows: pulley type, 3 tons per hour; drum type, 10 tons per hour; belt type, 16 tons per hour.

A wet magnetic separator operating on magnetite ore has been described in detail by Roche and Crockett.[10] This machine is a submerged-belt type and produces a high-grade concentrate with a recovery of practically 100 per cent of the magnetite. The capacity is given as 10 to 20 tons for a 36-in. magnet width.

Another wet separator, the magnetic log washer, has been described by Davis (reference of footnote 3). Capacities of 50 to 75 tons per day with a 2 by 14-ft. machine are given. The experimental concentration of roasted hematite on a magnetic log washer has been described by Lee, Gandrud and DeVaney.[11] When carefully operated the magnetic log washer will give an excellent product, as shown by the results obtained by Cooke in the re-treatment of a commercial magnetite concentrate (Table 5).

High-intensity Separators

High-intensity separators in use in this country are either of the induced-rotor or belt type. The use of an induced-rotor separator for separating feldspar and mica has been described by Burgess.[12] In this type of machine the material is passed over a laminated rotor, and as the stream falls off the rotor the magnetic material is deflected and falls in a different trajectory from the nonmagnetic material. This type of machine will make very good separations, but the maximum capacity of a machine is only about 2 tons per hour.

The Wetherill high-intensity separator has been used for years to separate products of rather low susceptibility. It consists[13] essentially of a feed belt passing between poles of strong electromagnets. A flat pole directly below the belt is opposed in polarity to a wedge-shaped pole directly above it, so constructed that a thin cross belt running in contact with its lower surface is slightly above the upper surface of the feed belt, so that ferromagnetic matter is removed from the feed belt in a direction at right angles to that of the feed movement. The magnets for the larger machines are wound for 30,000-amp., 60,000-amp. and 100,000-amp. turns, and may draw as much as 35 amp. at 110 volts and consume 1½ hp. for mechanical operation. The capacity may be as low as ¾ ton per 24 hr. on wolframite-cassiterite ore and as high as 4½ tons or more per hour when removing franklinite. Discussions of the performance of these machines on various ores are readily accessible.[14]

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[10] H. M. Roche and R. E. Crockett: Magnetic Separation. An Up-to-Date Mill. *Eng. & Min. Jnl.* (1933) **134,** 273.

[11] O. Lee, B. W. Gandrud and F. D. DeVaney: Magnetic Concentration of Iron Ores of Alabama. U. S. Bur. Mines *Bull.* 278 (1927).

[12] B. C. Burgess: Methods and Costs of Milling Feldspar at the Minpro Plant, Tennessee Mineral Products Corporation, Spruce Pine, N.C. U. S. Bur. Mines *Inf. Circ.* 6488.

[13] For description, see R. H. Richards: Ore Dressing, **3,** 1521–1525. New York, 1909, McGraw-Hill Book Co.

Another type of high-intensity separator has been used in Europe, and has been described by Granigg.[15] Since his article is not generally available, the development is described in some detail here. The separator consists essentially of a conveyor screw (Fig. 8) of soft magnetic material, which is part of a closed circuit in iron interrupted only at the working space (a, b, c). Two coils, w_1 and w_2, situated on the shaft L generate a magnetic flux flowing through the shaft and conveyor screw.

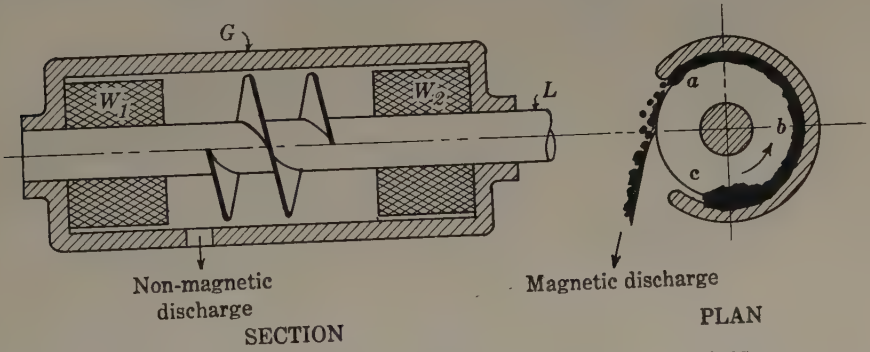

Fig. 8.—Schematic sketch of screw-conveyor separators.

The magnetic material adheres to the screw in the concentrated field between the thread and the casing g and is carried upward as the screw rotates and discharges at an opening in the casing assisted by a brush. The nonmagnetic material falls by gravity through an opening in the lower part of the casing. The field is given as more than 12,000 oersted. An advantage claimed for this type of separator over the belt and ring types is that the material is in actual contact with the magnet poles from the beginning, hence no lifting through an air gap is necessary. Furthermore, the material to be separated is pushed forward by the screw and is subjected to a repetition of magnetic action at each revolution of the screw. The machine can be used either dry or wet. A few examples of application are shown in Table 6.

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[14] R. V. Ageton: Milling Manganese Ore at Algonquin Mine, Philipsburg, Montana. *Eng. & Min. Jnl-Pr.* (1923) **116,** 181–183.

W. E. Hitchcock and J. R. Pound: Magnetic Separation of Bismuth, Tin and Tungsten Concentrates in Tasmania. *Min. & Sci. Pr.* (1920) **120,** 379, 382.

R. H. Richards: Reference of footnote 13.

A. F. Taggart: Handbook of Ore Dressing, 921–923. New York and London, 1927. John Wiley & Sons and Chapman & Hall.

H. A. J. Wilkins and H. B. C. Nitze: The Magnetic Separation of Non-Magnetic Material. *Trans.* A.I.M.E. (1897) **26,** 351–370.

[15] B. Granigg: *Erzbergbau, Erzaufbereitung und Metallhüttenwesen* (1931) **1,** 75.

TABLE 6.—*Examples of Magnetic Concentration with the Granigg Separator*

Current, Amp.	Ore		Composition of Products, Per Cent		Recovery, Per Cent
	Description	Composition, Per Cent	Magnetic	Nonmagnetic	
0.75	Quartzitic hematite	Fe, 37; SiO_2, 45.4	Fe, 53.5; SiO_2, 30.8		90.7
0.4–0.6	Hematite and magnetite		Fe, 65; SiO_2, 3–4	Fe, 7.3; SiO_2, 75.26	
1.5	Chamosite, unroasted	Fe, 25	Fe, 35.6	Fe, 5.6	90
	Siderite and hematite; tetrahedrite and chalcopyrite			Cu, 20	
2.0	Manganese (pyrolusite)		Mn, 49	Insol., 85; Mn, 8.57; Fe, 0.14	
	Quartz, feldspar, garnet, phlogopite, etc.			Quartz feldspar, Fe, 0.2	
0.4	Leucite-phonolith contaminated with iron	Leucite, 25		Fe, 0.4	
	Nephelite sand		Eudialite, FeO, 5.5		
	Silicic acid (chemically pptd.)	Fe, 0.2		Fe, 0.02	
	Coal, low fusing point ash		Ash-producing silicates, Fe, 1.0	Coal with high-fusion-point ash	

Data on the capacities of machines of this type are not available, but the fundamental difficulty in high-intensity separation, the necessity of passing the material through a narrow gap to insure high concentration of the field, has not been overcome, hence capacities will be low compared to those of low-intensity machines.

NEW SEPARATORS BASED ON COERCIVE FORCE AND REMANENCE

Earlier in the paper it was pointed out that the coercive force and remanence of magnetic minerals are amenable to control by physical and heat treatment. Methods of magnetic separation have been devised that depend on these properties rather than on the usual mass susceptibility.

If a mineral with high coercive force and remanence, such as reduced hematite, is subjected to a magnetic field it will be permanently magnetized. Each particle will become a permanent magnet and will adhere to the other particles. Reduced hematite can be separated from a low-coercive-force material, like magnetite, or a nonmagnetic material, like sand, by magnetizing and screening. The low-coercive-force and nonmagnetic material will pass through the screen, while the reduced hematite will agglomerate and remain on the screen.

A more useful method of separation and one that is applicable to materials of lower remanence depends on the behavior of a high-coercive-force particle in an alternating magnetic field. The hysteresis loop of a high-coercive-force mineral particle, as in Fig. 9, after preliminary magnetization becomes a small bar magnet with poles of intensity $+a$ and $-a$. If it is subjected to the action of a magnetic dipole, the adjacent poles of the particle and the dipole being of opposite sign, represented by $+b$ and $-b$, it will tend to be demagnetized to the values $+c$ and $-c$ but will be oriented so that it is attracted to the magnetic dipole. If the polarity of the magnetic dipole is reversed, the mineral particle will be repelled with a force proportional to the product, intensity of the dipole b and intensity of the small permanent magnet c. The repulsion from a surface accordingly will be given by the formula $\frac{bc}{d^2}$, where d is the distance from the magnet pole and the best conditions for repulsion will be $\frac{bc}{d^2}$ = maximum. For most minerals the curve ay is practically a straight line, so that the maximum product of c and b occurs when the field used is exactly one-half the coercive force. The repelling force then will be proportional to $\frac{1}{4}H_cI_r$.

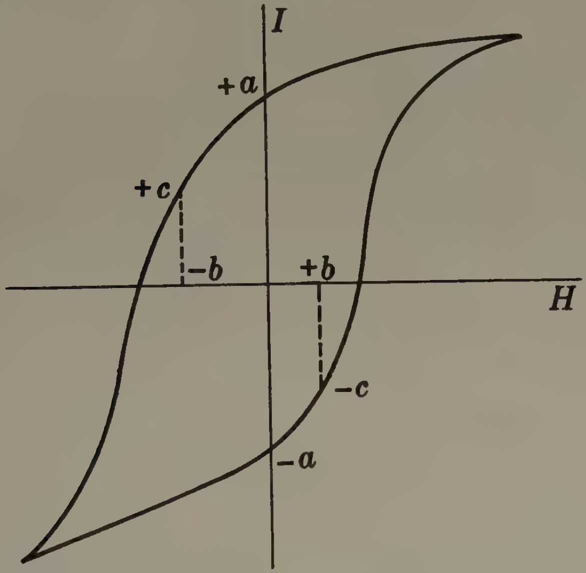

FIG. 9.—HYSTERESIS LOOP OF MINERAL WITH HIGH COERCIVE FORCE.

Under these conditions an alternating magnetic field causes the particles to be repelled whenever their polarity is opposed to that of the field, to be attracted when in unison with the field, and to be rotated when in any other position. During movement by repulsion or rotation, they will obviously be freed from friction, so that if they are supported on a tilted surface by friction the action of an alternating field will cause them to proceed rapidly down the plane. Such action on the part of the high-coercive-force particles can readily be used for separation purposes. First, however, the effect of the frequency of the alternating field on the behavior of high-coercive-force particles in fields of various intensities will be considered. As has been seen, the magnetized particles stick together

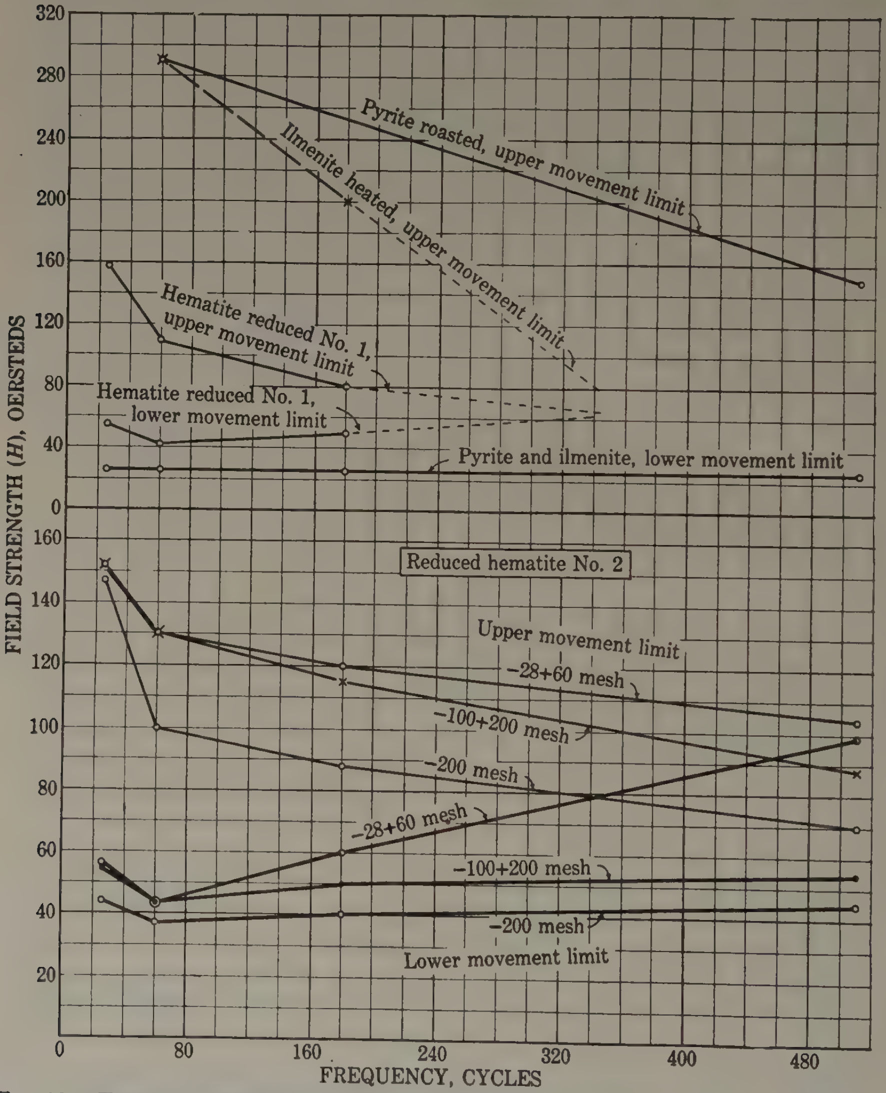

FIG. 10.—EFFECT OF FREQUENCY AND FIELD STRENGTH ON MOVEMENT OF MAGNETIZED PARTICLES IN ALTERNATING CURRENT FIELDS.

in the absence of external fields. As an alternating field of moderate frequency is applied and increased, this attraction of the particles for each other must be overcome. Expressed magnetically, with reference to Fig. 9, the impressed field strength, $+b$ and $-b$, must reduce the intensity of magnetization of the particles to a point where the orienting force of the applied field is greater than the force of the particles themselves. That is, b must be numerically greater than c. Materials with relatively low remanence and high coercive force are, therefore, best suited to activity in an alternating field. As the field is further increased, we have seen that the repelling force passes through a maximum at $\frac{1}{2}H_c$ and then decreases to zero in the neighborhood of H_c. The effect of frequency is first to break up the mass movement of groups of particles as it increases from zero to about 25 cycles, then as it increases further to increase the field necessary to overcome the initial stickiness and decrease the field at which the activity becomes substantially zero; that is, to narrow the range of field strength in which the repelling action is evident. Fig. 10 shows the relation between the field strength, frequency, and upper and lower limits of activity in an alternating magnetic field. Whether the effect of frequency is a purely mechanical one due to the inertia of the particles or an actual effect on the magnetic properties remains to be determined.

CONSTRUCTION AND OPERATION OF SEPARATORS

A great many types of separators have been built in the laboratory utilizing the principles outlined herein. Of these the authors have selected two as best suited to commercial work. These are the belt separator and the drum separator.

Belt Separator.—Fig. 11 shows one design for a commercial machine with a capacity of 10 to 20 tons per hour. The characteristics are carefully extrapolated from tests on a laboratory-size machine having a single belt. The machine as shown has two magnets of different strength. The number of magnets may be increased with a continuous variation in field strength. Counter poles above the belt may be used if desired to increase the field strength. In general, a field strength of 50 to 300 oersted is used.

The method of direct-current activation preliminary to separation is not shown but in most instances may be accomplished by simply passing the feed over a relatively strong direct-current magnet. Where strong activation is necessary, as in untreated hematite, the material is passed between magnet poles in the form of toothed cylinders, so that every particle is subjected to a high direct-current field.

Drum Separator.—Fig. 12 shows one design for a commercial drum separator with a capacity of 5 to 8 tons per hour. This separator is compact and gives very high capacity per unit of magnet surface.

PRACTICAL APPLICATIONS

In addition to the quantitative results given in Table 7, a few others are informative.

Treatment of Magnetite Ores.—Some magnetites have sufficient coercive force to enable their separation by alternating current and others may be heat-treated to condition them for such a separation. It would seem better, however, to treat ore containing its iron as magnetite by direct-current machines.

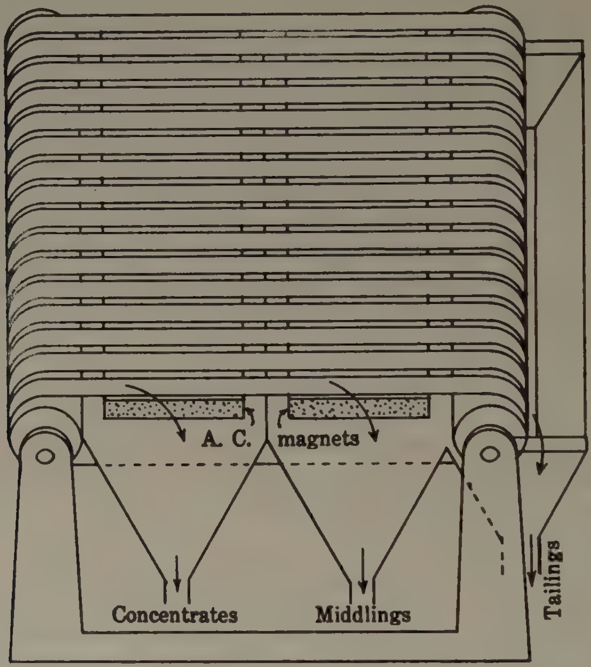

FIG. 11.—SCHEMATIC SIDE VIEW OF BELT-TYPE H_c SEPARATOR.
Capacity 10 to 20 tons per hour. Sixteen belts, 3 ft. × 3 in. upper surface, 15° to 40° slope. Space between belts, 1 in. Belt travel, 20 ft. per min. Pulley, 6-in. dia. Size over-all 5 by 5 by 4 feet.

Treatment of Manganese Ores.—Most manganese minerals may be made active with alternating current by suitable heat treatment, so that there may be a place for separation by alternating current in this field.

Ilmenite Ores.—Ilmenite and titaniferous magnetite are made active by a roast at 700° to 900° C. and may in consequence be separated by H_c machines from all substances such as gangue, pure hematite, magnetite and the other usual constituents of black sands.

Differential Separation of Ilmenite and Magnetite.—The difference in magnetic properties of magnetite with varying TiO_2 content permits the separation of concentrates of titaniferous magnetite into their mineralog-

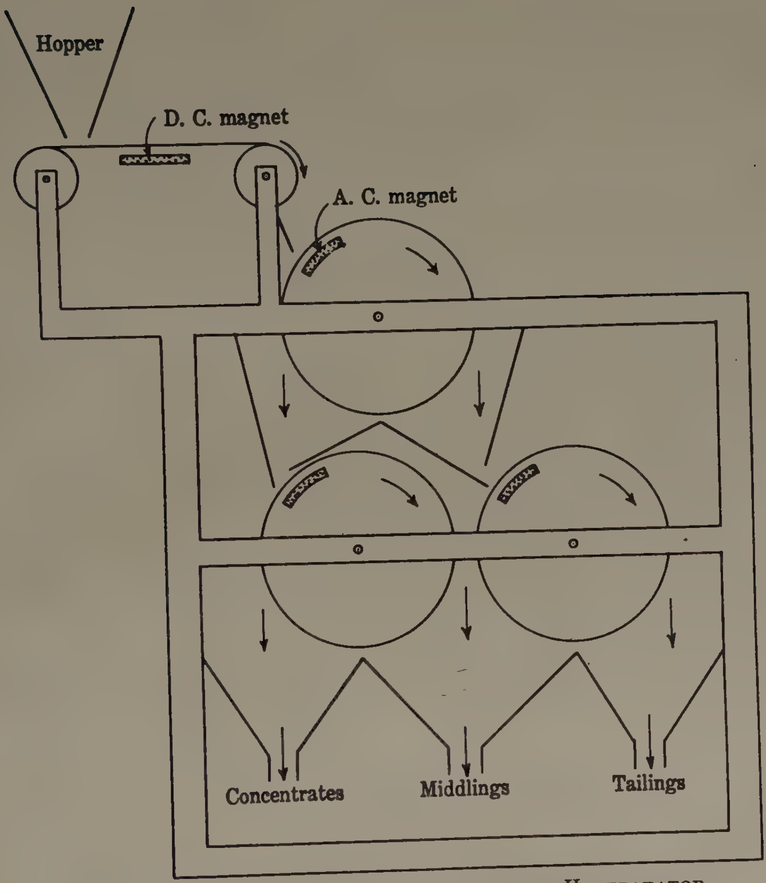

FIG. 12.—SCHEMATIC SIDE VIEW OF DRUM-TYPE H_c SEPARATOR.
Feed capacity, 5 to 8 tons per hour. Drum diameter 1 ft., length 8 ft., r.p.m. 2 to 5. Size over-all, 4 by 5 by 10 ft.

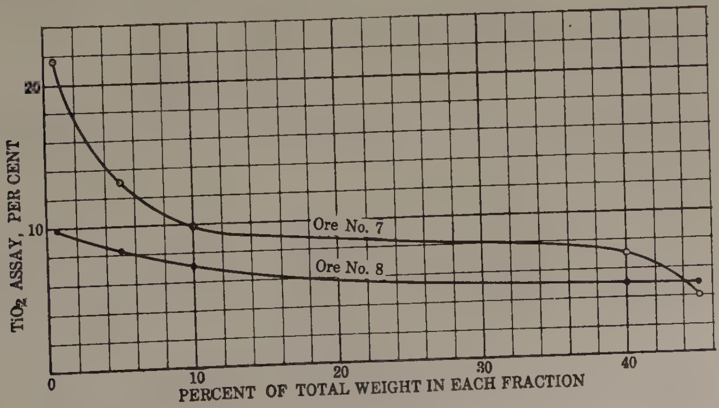

FIG. 13.—DIFFERENTIAL SEPARATION OF TITANIFEROUS MAGNETITE.

ical constituents by the H_e separators. Two samples of titaniferous magnetite were separated into a number of fractions by using a variation of field intensity. The results on ore No. 7 and ore No. 8 are shown graphically in Fig. 13. The particles of these ores range in composition from 4 to 21.7 per cent TiO_2, and by proper adjustment of the separator a split can be made at any point with, of course, the recovery indicated by the ore composition.

TABLE 7.—*Examples of Magnetic Concentration with Separators Using Alternating Current*

Ore	Treatment	Products		Recovery, Per Cent
		Description	Composition, Per Cent	
Limonite in barite, intergrown, 50 per cent Fe	Crushed −28 mesh; flash roast 1400° C. Separated on belt-type a.c. machine after activation.	Concentrates	Fe, 57.88	Fe, 90.4
		Middlings	Fe, 38.6	Fe, 7.0
		Tails	Fe, 6.62	Fe, 2.6
Limonite in barite, intergrown, 50 per cent Fe	Crushed −28 mesh; flash roast 1400° C. Separated on drum-type a.c. machine.	Concentrates	Fe, 61.44	Fe, 85
Iron ore magnetically roasted, 38.0 per cent Fe	Oxidizing roast 5 min., 600° C. Separated on belt-type a.c. machine.	Concentrates	Fe, 60.2	Fe, 81.8
		Middlings	Fe, 38.3	Fe, 14.0
		Tails	Fe, 7.7	Fe, 4.2
Iron ore magnetically roasted, 38.0 per cent Fe	Oxidizing roast 5 min., 600° C. Separated on belt-type a.c. machine.	Concentrates	Fe, 60–61	Fe, 90[a]
Waste product, Rheolaveur plant, 35.0 per cent Fe	Crushed −28 mesh, standard magnetic roast, a.c. separated.	Concentrates	Fe, 65.4	Fe, 94.8
		Middlings	Fe, 6.0	Fe, 4.2
		Tails	Fe, 1.3	Fe, 1.0
Hematite ore, 48 per cent Fe	High-intensity activation, a.c. separated.	Concentrates	Fe, 66.1	Fe, 84.6
		Middlings	Fe, 42.4	Fe, 10.3
		Tails	Fe, 5.3	Fe, 5.1
Chrome-iron ore, olivine gangue, 50.46 per cent Cr_2O_3	Crushed to −100 mesh, oxidizing roast 900° C., standard magnetic roast, a.c. separated, belt-type machine.	Concentrates	Cr_2O_3, 59.1	Cr_2O_3, 79.6
		Middlings	Cr_2O_3, 47.0	Cr_2O_3, 16.5
		Tails	Cr_2O_3, 12.0	Cr_2O_3, 3.9
Chrome-irone ore, 35 per cent Cr_2O_3	Crushed to −100 mesh, oxidizing roast 900° C., standard magnetic roast, a.c. separated, belt-type machine	Concentrates	Cr_2O_3, 63.3	Cr_2O_3, 67
		Middlings	Cr_2O_3, 19.0	Cr_2O_3, 30
		Tails	Cr_2O_3, less than 5	Cr_2O_3, 3
Ferberite, 25 per cent WO_3	Oxidizing roast 950° C., then reduced 600° C., crushed to 28 mesh, a.c. separated.	Concentrates	WO_3, 61.0	WO_3, 73.2
		Middlings	WO_3, 19.8	WO_3, 23.8
		Tails	WO_3, 1.9	WO_3, 3.0
Cobalt ore, 1 per cent Co	Reduced in CO at 300° C. 30 min., crushed to 60 mesh, a.c. separated.	Concentrates	Co, 21.2	Co, 73.5
		1st middling	Co, 3.86	Co, 17.6
		2nd middling	Co, 0.30	Co, 5.1
		Tails	Co, 0.11	Co, 3.8
			Fe Cu Zn	
Residue from chemical plant. Reported to contain Fe_2O_3, 60 per cent; FeS, 9; $ZnSO_4$, 1.5; CuO, 0.75; SiO_2, 8.0; Fe-Ca-Mg-SiO_2, 17	A.c. separation, no pretreatment except crushed to −40 mesh.	Concentrates	63.19 0.66 0.97	Fe, 65.8
		Middlings	57.41 0.39 0.87	Fe, 14.9
		Tails	27.59 0.21 0.68	Fe, 19.3
Hematite, 1.6 per cent S	Heated 500° C. in closed container. Activated and a.c. separated.	High H_e	S, 0.82[b]	Fe_2O_3, 70.6
		Low H_e	S, 2.3	Fe_2O_3, 29.4
Hematite, 1.6 per cent S	Slight reducing roast, a.c. separated.	High H_e	S, 1.17	Fe_2O_3, 93
		Low H_e	S, 4.58	Fe_2O_3, 7
Auriferous pyrite	Strong oxidizing roast, a.c. separated.	High H_e	1.49 oz. Au per ton	Not good
		Low H_e	0.29 oz. Au per ton	
Pyrite with siliceous gangue	Crushed to −28, flash-roasted at 600° C.	Concentrates	Fe, 50.1	Fe, 79.3
		Middlings	Fe, 10.8	Fe, 6.8
		Tails	Fe, 2.0	Fe, 13.9

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[a] The present method of treatment for this ore (Gröndal drums) is said to result in an 85 per cent recovery in the form of a 56 per cent product.

[b] This sample had failed to show any segregation of sulfur by d.c. magnetic separation or by various other ore-dressing treatments.

Tungsten Ores.—Ores containing tungstates of iron and manganese may have the susceptibility, coercive force and remanence of their tungsten minerals increased markedly by a reducing roast. The activity is still further increased if a treatment with sodium hydroxide or an oxidizing roast precedes the reduction.

SUMMARY

A review of the patent literature indicates that practically all known magneto-mechanical effects have been proposed as the basis of magnetic separation. Commercial separators nevertheless have utilized only tractive forces. It is suggested that this is due to the paucity of reliable data on the complete magnetic properties of natural and treated minerals. Complete magnetic data are given for the first time for a number of important minerals, and the application of these data to problems of magnetic separation is discussed.

The field and limitations of existing commercial separators are discussed, and a new type of separator based on repulsion rather than attraction is described. A few results obtained with the new type of machine are given.

Economical Techniques in Treatment of Gold Ore

By A. W. Allen,[*] Member A.I.M.E.

Progress in the art of amalgamation in recent years has been negligible, partly because a copper plate, though it occupies extensive floor space, requires frequent attention and invites theft, is a simple piece of apparatus that gives visible results; partly because the impression prevails that amalgamation is too primitive a process to merit consideration by an up-to-date metallurgist. Both these objections can be overruled. Plates can and will be superseded by more efficient, theft-proof amalgamating units. Improved methods of application other than by settlement or by impact, neither of which is effective for removal of fine gold, will be developed, and the process given wider opportunity. As A. F. Crosse aptly remarked, neglect to utilize amalgamation when free gold can be recovered thus is comparable to the decision of a storekeeper to deal only with credit customers.

Usual practice is to follow amalgamation with cyaniding. Rarely is attention paid to the possibility of reversing the order—of extraction of the fine gold first by cyaniding; the coarse, subsequently, by amalgamation. Such a sequence avoids the alleged hazard that amalgamated gold particles may be unacted upon by cyanide, whereas cyanided gold particles are readily amalgamated. Research has indicated, however, that the mercury coating on a gold particle is easily soluble in cyanide solution, probably because such coating is a compound of gold and mercury. Comparative-test results at one plant, of cyaniding a ground concentrate before and after amalgamation, showed total extractions of about 90 and 100 per cent, respectively, indicating high solubility of the mercury-gold particle, if present, or the advantage of preliminary impoverishment before cyaniding, or both. The contention that a loss in residue from an amalgamation-cyanidation plant is occasioned by presence of amalgamated gold that escapes action by solution is not supported by adequate evidence. The function and scope of mercury as an aid to extraction of gold by cyanide is deserving of attention. It is a powerful desulfurizer.

To restrict an extractive process to a concentrated product is logical and economical, if preliminary concentration will segregate a high per-

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centage of material treatable by such process. Amalgamation of the product of blanket strakes, instead of amalgamating the entire mill pulp, involves less expense, permits more precise control and the introduction of refinements in adjustment. Similarly, the cyaniding of flotation concentrate results in a high extraction from a small bulk of material. Such trends indicate the importance of studying the economics of metallurgical treatment and the mechanics of mineral separation.

Justification for impoverishing a gold ore before cyaniding is begun has been questioned so persistently, although generally in broad derogatory terms, that specific evidence of comparative results is of interest. For instance, a fine-ground pyrrhotite-aplite ore was given a straight all-cyanide treatment with agitation for 17 hr., involving a residue loss of about 17 grains gold per ton. After extraction of 75 per cent of the gold in the same ore by amalgamation, a cyanide treatment of the tailing, with agitation for only 6 hr., resulted in a residue loss of under 10 grains per ton.

Such comparative results emphasize the necessity for distinguishing between the terms "extraction" and "recovery," the former referring to a preliminary phase only of a gold-recovery process. Losses in retorting and melting amalgamated gold are negligible, so "recovery" is almost analogous to "extraction." Extraction by cyanide is seldom approached and never equaled by recovery. Extraction by gravity concentration or by flotation is far removed from recovery. Losses by imperfect displacement of solution by wash are inevitable in the mechanical treatment of a pulp in any process involving chemical dissolution. Recovery of gold by cyanide may also be adversely affected by absorption or adsorption of the metal, or by both. Hydrometallurgical methods of treating concentrate to produce bullion involve mechanical loss. If such concentrate is sold to the smelter, an expense for transportation and treatment must be met. Agreement as to amount due is rare, involving loss or additional cost for umpire assaying and duplicated sampling, with deduction from current gold value in the ultimate settlement.

Determination of percentage extraction of gold by concentration or cyaniding is as valuable and as necessary in plant practice as it is in preliminary work; but the superiority of the technique of treatment should be gaged by actual recovery in bullion or its cash equivalent. To distinguish between the terms "extraction" and "recovery" is the first step in planning satisfactory economic result.

Bulk Leaching of Low-grade Ores

Recommendation that bulk-leaching tests, after comparatively coarse crushing, be made on an ore apparently pervious to solution, in which the gold was admittedly fine, was dismissed with the information that such a method had been tried and abandoned a quarter of a century before; to which a retort was made that Chuquicamata was then a mountain of discolored stone, to be subsequently transformed, mainly by application of a new technique of bulk leaching, into one of the world's great copper mines.

Economic necessity is forcing attention to simple and inexpensive methods of gold recovery. Metallurgists are daring to go back to fundamentals, to risk the scorn of being stigmatized as out of date in their recommendations. Successful application of bulk leaching at several properties in recent years, on raw ore and on tailing, indicates that deposits of leachable material have been neglected, because of concentration of attention on fashionable metallurgical trends; that small profit by a simple and inexpensive process is preferable to financial loss by a complicated and costly one. The principal precaution in adopting bulk leaching is to remember that methods for distribution of the charge in the vat of a high-slime gold ore should not be patterned on those adopted for similar distribution of a low-slime copper or gold ore. High tailing loss after bulk leaching usually is the result of imperfect and uneven filling of the vat, of short circuiting of solution.

Excess of fines in a leach charge causes high resistance to solution flow, and a "break-through" may occur as pressure develops. If the slime is in moderate amount and evenly distributed, its treatment by gravity leaching provides ideal conditions for dissolution of gold and displacement of solution. The complacency discernible in reports that disclose erratic extraction because of short-circuiting of solution is not justified. The cause is not irremediable.

Physical effects of milling an ore in excess of water are not generally realized. I was recently reminded of an account of leaching operations that appeared in a metallurgical textbook. The ore was crushed to 30 mesh, delivered into a vat already half filled with lixiviant. "It was found," plaintively records the operator, "that the solution would not filter." This incident would not be mentioned if such a method of attempting to leach a sand-slime charge were not being repeated. Some technicians fail to realize that during milling in water, or by delivery of dry-crushed ore into water or solution, a separation inevitably occurs of colloidal from crystalline matter, of coarse particles from fine ones, of light mineral from heavy mineral, inhibiting a redistribution of constituent elements that, if possible, would permit even percolation of leach solution. If the filter-aid properties of the crystalline material are to be utilized to effect lixiviation of the amorphous material, without recourse to a greater pressure or vacuum than that obtainable by gravity flow, the ore must be distributed in the dry state, or it must be dried after wet milling—a step that in several instances has been proved economically practicable.

TIME FACTOR IN HYDROMETALLURGICAL TREATMENT

Rarely given adequate consideration by those planning a treatment method are the comparative merits of short or long contact of ore with solvent solution, when prompt treatment is not technically essential. Time is the cheapest of "commodities." An extraction unit consisting of leaching vats and circulating pumps involves minimum capital and operating outlay. Indicative data on the solubility of gold show that a high extraction of minute particles (minus 260 mesh) is generally effected in the normal time available for agitation and filtration treatment in an all-slime plant; whereas an almost complete extraction is obtainable of minus 60-mesh gold if contact is maintained for several days. Heap-leaching of uncrushed copper ore has demonstrated that lengthy treatment at small cost may be profitable, whereas an expensive quick treatment of the same ore finely ground would be economically impracticable.

Solubility of gold of any size can be hastened by fine grinding in contact with cyanide solution; but with low-grade ores this is profitable only on a concentrated product, thereby avoiding the cost of sliming much barren gangue and the complications involved by its enrichment with valuable solution. Expense and efficiency of solution recovery are details of operations sometimes overlooked after tests have been made to determine extraction of gold by sliming-agitation treatment, especially when comparing such data with similar laboratory or trial results obtained by leaching a sand that can be easily and thoroughly impoverished during displacement washing by gravity flow. Opportunity for cyaniding at rest a sand product not ground to the fineness needed for a hurried agitation and filtration treatment should merit consideration.

Adoption of all-sliming in solution, followed by agitation of the entire pulp, has been justified by results at many plants; but instances are on record to prove that the alternative—an extractive treatment by gravity leaching—has not been investigated. No patented machinery is necessary, and the incentive to wide technical publicity and organized recommendation is therefore absent. Knowledge of fact comes mostly from disclosure of experience by a busy metallurgist on an operating property, who is concerned solely with economical processing and technical adequacy. Thus, an important contribution to the subject of leaching has been made by Mr. Allan J. Clark, of the Homestake company, who reports that, after efficient desliming of the pulp, one-third of the weight of a sand charge at Lead is in the form of minus 200-mesh material, and that an excellent extraction of the gold in this portion is obtained by gravity leaching. After removal of all the slime by cone classification, the exceedingly fine crystalline material offers no resistance to even passage of solution and successful impoverishment of the charge. An important advantage of gravity leaching, seldom recognized, on a bulk product or on a separated sand, is that a progressive enrichment of solution occurs simultaneously with a progressive impoverishment of the charge—indicating ideal conditions for efficient extraction.

Double treatment of sand and slime, as practiced at Lead and in South Africa, is usually considered unnecessarily complicated for adoption in a new plant; but results from both these widely separated districts indicate that metallurgic efficiency is attained thus at low operating and amortization cost. Instances are on record to show that an unsatisfactory extraction of gold from comparatively coarse sand by gravity leaching in the laboratory has led to the abandonment of interest in the method, to substitution of grinding to minus 200 mesh and finer, whereby an almost complete extraction is obtained. Rarely do recoveries in practice approach such extraction results. Efficiency of displacement of dissolved gold from wet pulp decreases as the ore is ground finer. This is partly because of lessened permeability of the ore mass as compression occurs during vacuum or pressure filtration, partly to the vast increase of surface area exposed by comminution, leading to physicochemical action that exerts a disturbing influence on the stability of a solution containing cyanide and a precious metal. Natural slime has been shown to be capable of absorbing a gold solution during milling in cyanide, to be subsequently displaced, usually in part only, by slow osmotic action during washing. Excessively fine grinding also facilitates adsorption of gold, which may explain the difficulty experienced in reducing by ordinary methods the gold content of washed residue. Post-treatment with a solution that disturbs the adsorption equilibrium and releases the adsorbed gold is indicated as a remedy.

Need for such a refinement in metallurgical practice, however, deflects attention to the mistake of applying remedial measures, which are costly, at the end of operations, instead of avoiding the cause of high residue. Superfine grinding of valueless gangue in contact with an unstable solvent in which gold is dissolved, when unnecessary for extractive purposes, invites hazard. Experience is showing that, when such grinding is necessary, it is preferably done in highly alkaline water, to counteract cyanicide action, effect coagulation of slime particles and insure maximum adsorption of valueless instead of valuable ions.

The strangest sequel to the development of efficient and economical dewatering devices is that they have been applied extensively at the tail end of the cyanide process, in an effort to reduce residue loss by repeated repulpings and filtrations, instead of for dewatering pulp before cyaniding is begun. No generalization is intended by the statement that on silver ores in Mexico and on gold ores in Australia, Africa and South America, I have demonstrated to my own satisfaction that high residue after milling in cyanide is usually avoidable by change of procedure—by milling in alkaline water, a change seldom anticipated when an all-slime plant is designed, hence rarely effected.

Reawakened interest of late in the advantages of milling a gold ore in water or lime water instead of in cyanide solution is gratifying to those who attempted to encourage unbiased discussion of the subject when the "new metallurgy" became the vogue 15 years ago. Whether the logic of removing coarse gold by amalgamation before cyaniding is begun is admitted or denied, whether destruction of cyanicides and pre-aeration and coagulation of slime particles is favored or not, milling in water is now an essential preliminary to flotation, giving opportunity for prior recovery of coarse gold by amalgamation and for the discard or separate treatment of slime, the presence of neither of which is conducive to efficient flotation results.

Experience on low-grade ores, study of the history and the results of failures as well as successes, attention to the outcome of following fashion instead of considering each ore as a separate problem, indicate that general recommendation, even in the direction of the latest trend, flotation, is unwise. Low-grade ore is available that appears to be amenable, at small cost, to simple treatment methods, such as amalgamation, table concentration or bulk-leach cyaniding.

PREVENTION OR CURE OF METALLURGICAL COMPLICATIONS

Technical trends are stimulated by publicity; publicity is stimulated by commercial activity and investment. A satisfactory technique of ore treatment that involves use of unpatented or unadvertised apparatus rarely receives wide publicity or evokes extensive interest. For instance, the drying and heating of ore preparatory to hydrometallurgical treatment has received scant attention, although removal of moisture permits dry crushing and the advantages of utilizing methods of extraction that are cheap and effective. Heating destroys colloidity and amorphous characteristics, inhibits the action of absorbents, increases the friability of crystalline matter, reduces cost of comminution, simplifies subsequent settlement, thickening and filtering operations. Despite the comparatively high thermal efficiency of a properly designed drier, rarely are the advantages of this step in ore preparation considered when plans are being discussed. Wide publicity is accorded the results of avoiding the roasting of a refractory ore or intermediate product; much less attention is given to the benefits accruing from roasting, which include loss of weight, favorable alteration in chemical composition, removal of interfering elements, destruction of refractory constituents, and release of gold in a form usually recoverable by simple and inexpensive methods. G. H. Buerg[1] has demonstrated that application of an intensely hot flame to the surface of an ore that showed no gold under high-power magnification resulted in the appearance of the metal on the surface, in the form of ball or cauliflower-shaped aggregates—a discovery of metallurgical significance.

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[1] G. H. Buerg: *Metall und Erz* (1930) **27**, 333–338.

Object lessons in technical history are drawn but rarely, although details of cause and consequences of a fiasco, the recording of a disappointment, may prove as inspiring and educational and helpful as a colorful record of the direct or immediate antecedents to a dramatic success, and not less interesting. For example: A mining company for over two decades paid yearly dividends amounting to about 75 per cent of its capitalization. Metallurgical practice at the property comprised a simple roasting, dry-crushing and bulk-leaching treatment of an ore containing graphitic carbon. Prevailing metallurgical trends then suggested radical changes in processing, involving the substitution of ball milling in cyanide solution, hydrometallurgical treatment of the sand and slime products, and the roasting only of a concentrate that contained none of the graphite. A substantial reduction in cost of treatment was promised, more than sufficient to justify an expected higher loss of gold in residue. But extraction was reduced so seriously by the change that the company, burdened with the result of heavy capital expenditure for new plant and faced with diminished gold yield, ceased to pay dividends.

The technical explanation was simple: Although milling in cyanide solution insured early dissolution of the gold, it also facilitated early premature "precipitation" on the graphite, ground to a fineness that enhanced adsorptive action. Previously, roasting rendered the slime nonamorphous, and easily leachable by inexpensive gravity methods. The same product in a raw state, during the separate pressure or vacuum treatment essential after even moderate wet comminution, gelatinized to an extent that upset all calculations. The result of the changes deflected attention to the chemical and physical and mechanical advantages accruing from heating and roasting a refractory ore. It emphasized the fact that prevention of technical troubles is preferable to and cheaper than their cure by the most modern of remedies.

In essaying to explain an unrecoverable loss of gold in slime residue several years ago[2] I cited the fact that colloidal aluminum hydroxide was capable of absorbing gold from a colloidal solution. I suggested that the metal might occur in ore in that form, that colloidal clay might absorb it during hydrometallurgical operations.

Colloidal gold is usually considered to be in the form of a suspensoid, of such minute particle size that it is invisible except when occurring in quantity sufficient to color the surrounding liquid. It can be produced by the electrical disintegration of the metal, under water, but not by comminution. It is a deep red color when made chemically by action of sodium carbonate and formaldehyde on a dilute solution of gold chloride.

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[2] A. W. Allen: *Trans.* Inst. Min. and Met. (1915–16) **25,** 121–139.

Addition of dilute sodium carbonate solution changes the color to blue; addition of concentrated sodium carbonate solution, to brown. The dark precipitate obtained by action of zinc on aurocyanide solution probably contains colloidal gold.

Colloidity is destroyed by drying and application of heat. Presence of colloidal gold in a gold ore is assumed by many operators and investigators. If the assumption is correct, a heat treatment is the only method I can suggest to effect a change in physical condition of the gold, an exposure in the form of particles of appreciable size, that would permit its recovery by commercial methods.

Aeration of Cyanide Solutions

Interest is revived at intervals on the value of oxygen as an aid in dissolving gold by cyanide, new apparatus is devised for aerating plant solution, and Elsner's equation of 1842—providing an academic explanation of the union of gold, potassium cyanide, oxygen and water to form potassium-gold cyanide and caustic potash—is cited. Its validity is not questioned; but many competent chemists and metallurgists, including the senior inventor of the cyanide process, have insisted that gold is soluble in a cyanide solution bereft of oxygen, to which no oxidizing agent has been added. Other competent chemists and metallurgists, accepting the Elsner equation as adequate to explain all that occurs in laboratory test or in plant practice, are equally positive that oxygen is essential. The disparity of opinion indicates a common inclination to think and reason without consideration of concomitant chemical reactions, physical and electrical effects.

Julian, in 1892, patented a method for aerating working cyanide solutions and for the use of many chemical oxidizer compounds; but he also demonstrated that gold is soluble in such solution in absence of oxygen. These two facts demonstrate the wisdom of differentiaeing between laboratory demonstration and commercial practice. They deflect attention from any single chemical equation as an explanation of all the reactions that occur, and they emphasize the need to study factors and variables that an equation ignores. In reviewing divergent professional conclusions on the function of oxygen in dissolving gold in ore, I am reminded of comments I made a few years ago. I am reported to have said[3] that "we have been prone to consider the composition of a mineral substance according to chemical analysis, forgetful of the fact that the condition of the surface and the character of what is adsorbed thereon constitute powerful factors on the results of any treatment involving delicate or initial reaction."

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[3] Quoted by O. D. Welsch: *Eng. & Min. Jnl.* (1932) **133.**

These remarks might be slightly paraphrased to account for the conflicting opinions as to the function of oxygen in the dissolution of gold by cyanide. Pure gold of uniform surface characteristics is doubtless insoluble in a pure solution of cyanide in oxygen-free water; but perfect purity and absolute uniformity are attainable and maintainable only by delicate manipulation and precise control in the laboratory. A minute degree of contamination is sufficient to cause galvanic action that stimulates active dissolution. Gold in cyanide solution in a glass vessel is only slightly attacked; dissolution becomes active if it is put in contact with a piece of iron. But such action diminishes unless the iron surface remains exposed, unless polarization is prevented; and oxygen is entirely effective for this purpose. Elsner's equation ignores galvanic action and subsidiary chemical changes that exert an important influence on the commercial extraction of gold by cyanide. It makes no mention of the formation of hydrogen peroxide as an intermediate product, as demonstrated by Bodlaender.

Pyrite, a common constituent of gold ores, provides a desirable contaminant that is electronegative to gold. Julian, who pioneered in the aeration of plant solutions 40 years ago, demonstrated this galvanic action by connecting a piece of gold to a piece of pyrite, both immersed in cyanide solution, through a galvanometer. An electric current was recorded as the gold dissolved, but its intensity rapidly decreased because of the deposition of hydrogen on the surface of the pyrite. Then, to satisfy conditions assumed by the Elsner equation, the gold was surrounded by air bubbles, then by oxygen bubbles, without favorable effect on current flow or rate of dissolution. When the air or oxygen was applied to the pyrite, however, immediate union with the hydrogen occurred, and maximum galvanic action and chemical activity was resumed. Complementary evidence that an element or compound electronegative to gold stimulates dissolution is seen in the fact that cyanide solution may penetrate a sulfide particle and attack encased gold.

Water, cyanide and oxygen do not comprise all the essentials for the commercial extraction of gold from ore. No single chemical equation is adequate to indicate all probable reactions. One function of the oxygen is to unite with hydrogen. The major effect, after permitting maximum galvanic action and maximum dissolution, is to reduce mechanical loss of cyanide, probably in the form of hydrocyanic acid gas. This economy is most noticeable when the gas is absorbed without agitation of the solution. An article recently published[4] begins by a recital of Elsner's equation and concludes with the statement that the most noticeable effect of extra aeration of solution in a series of large-scale tests was a substantial reduction in cyanide consumption.

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[4] Cyaniding Makes Progress. *Eng. & Min. Jnl.* (1934) **135**, 298.

Several years ago, in Mexico, I was confronted with the problem of counteracting the effects of reducing agents in the ore, necessitating unusually elaborate oxidation of slime pulp as well as of solution. Inefficient results being obtained with a Pachuca vat, air was then introduced at the bottom of a paddle agitator without baffles—insuring maximum swirling motion. The air was thus broken up into an infinite number of minute particles, which were retained in quiescent contact with the pulp, by reason of its almost horizontal swirl, and made to travel for long distances before release from the surface, where they appeared as does the gas from a liquid charged under pressure. Escaping air was discernible only by close inspection. Extraction results were greatly improved.

Solubility of a large bubble of air in water or cyanide solution is slight. Capacity of solution pulp to absorb a gas decreases with its viscosity, which partly explains the high dissolving effect of well aerated solutions permitted to percolate through a bed of ore. Agitation vats that function by circulating pulp by means of air that is not "atomized," with violent local pulp movements, are wasteful of cyanide as well as air. Maximum opportunity is provided for the hydrogen-oxygen reaction if the ore particles are allowed to fall through a well aerated solution. Excellent extraction at low cost has been obtained from an ore containing pyrite by comparatively coarse crushing, followed by counter-current decantation with such solution in thickeners carrying only a small quantity of settled pulp.

Gold Value and Metallurgical Result

Twenty years ago, at a meeting of the Institute in Salt Lake City, I had the temerity to criticize use of the monetary dollar as a unit in reporting assays of gold ores, of intermediate products and tailing. A practice then merely lacking logic has rendered technical reports meaningless and uninformative since the recent decline in the gold equivalent of United States currency.

Many if not most promoters and engineers assume that the commercial value of ore is still proportional to its dollar value; that, for instance, ore assaying $2 in Roosevelt currency offers the same opportunity for exploitation as did ore with similar characteristics that assayed $2 in Hoover currency. This is not so. Hoover-currency $2 ore contained about 46 grains of gold per ton. Roosevelt-currency $2 ore contains only about 28 grains.

Commercial success of a gold-mining enterprise usually depends on technical success, which in turn depends on the amount of gold possible to recover as well as on the market value of the metal. Percentage metallurgical extraction of gold in any ore, other factors being constant, decreases with grade or gold content. A 46-grain ore may be treated profitably when the market price of the metal is $20.67 per ounce; a

28-grain ore may yield insufficient to meet expenses after the price has been raised to $35 per ounce. Although many new opportunities are afforded the gold miner by currency depreciation, and exploitation of low-grade ore stimulated and made practicable, increase in price of gold, contrary to prevailing and wide-spread impression, does not invariably effect a corresponding increase in reserves treatable at a profit, or cause proportional gain in gold output by permitting beneficiation of rock containing less gold at a higher dollar value—an impression fostered by continued use of the dollar as a unit of gold in ores and plant products.

Disinclination to change this practice is due mainly to the inappropriateness of the only alternative unit that appears to be acceptable in the United States—the troy ounce. Run of mine ore, mill feed and final mill products usually range in gold content from one-quarter ounce to a few grains per ton. A troy unit intermediate between the grain and the ounce—the pennyweight—corresponding closely with the Hoover dollar, is used extensively outside the Americas for reporting gold assays, the majority of which are nearer 1 dwt. than 1 oz., so that the former unit, with decimal subdivisions, has proved simpler and preferable. Corresponding exactness is possible with fewer figures. For example: 13.3 dwt. compared with 0.665 oz.; 1.5 dwt. compared with 0.075 oz.; 0.5 dwt. compared with 0.025 oz. Thirteen point three is terser and more informative than is six hundred and sixty-five thousandths; one and one-half, than seventy-five thousandths; one-half, than twenty-five thousandths.

Mental conception of a unit weight of gold in ore and mill products is usually the equivalent to the pre-NRA dollar, or about 1 dwt., not the Roosevelt dollar of about 0.02857 oz. Although influences against uniformity seem to be dominant, general use of the pennyweight (dwt.) for reporting gold assays of ore and mill products, except perhaps concentrate, would permit standardization of data from the principal gold fields of the world, thereby permitting immediate comparison of technical results.

Ore Treatment as a Factor in Small Gold-mining Enterprises

By Ernest Gayford,* Member A.I.M.E.

When the United States Government started buying gold at varying prices per ounce, set by the President and the Treasury Department, the gold miner found himself facing new conditions, as gold became a commodity with a fluctuating market price. While it was self-evident that the miner was to receive more for his gold than the old established price of $20.67 per ounce, there was an element of uncertainty, which made it difficult, and perhaps dangerous, to decide to invest his time and money to produce gold, especially if the profit would represent only the difference between $20.67 an ounce and the new price as set from day to day by the Government.

When, however, on Jan. 30, 1934, the United States went off the gold standard, the price of newly mined gold being rather definitely set at $35 per ounce, with some assurance that the price would be maintained at or near this figure for a long time to come, the gold miner was justified in revaluing his property on the basis of the new price, and the prospector was offered inducement to spend his time and energies hunting for new mines, or going back to old locations previously abandoned as of doubtful value.

It is interesting to see what this new price means to the producer of newly mined gold, and how much of the increase is passed on to the producer. It will be found that, as with the base metals, the nearer he can get to producing a finished product himself the greater his reward will be; the net returns for bullion being necessarily higher than for the same amount of gold shipped in concentrates to the smelter.

Bullion can be shipped to the United States mints or Government assay offices under the following terms and conditions.

An affidavit must accompany the shipment, stating, among other things:

1. The source of the gold; whether ore, tailings or placer.
2. The tons of ore, tailings or cubic yards of gravel from which the shipment was recovered.

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3. The period within which the gold was taken from the mine or placer deposit.

4. The name and location of mine or placer deposit from which the gold was recovered.

Several other questions must be answered to prove that the bullion being shipped contains newly mined gold within the Act of March 9, 1933, and in compliance with the Gold Reserve Act of 1934. The necessary blanks for this affidavit can be obtained by applying to the nearest U.S. mint or Government assay office.

The consignment must consist of at least two troy ounces of pure gold with an assay fineness of at least 200 parts of gold out of 1000. The mint charges $1 for melting up to 1000 oz. or fraction, and 10¢ for each additional 100 oz. or fraction. There is also a treatment charge based upon the amount of base metal contained, varying from 1¢ to 8½¢ per ounce. Also there is deducted 0.25 per cent of the value to cover handling charges.

The express and insurance for shipping to the nearest U.S. mint or assay office would average about $2 for each $1000 of value, with a limit of 4 lb. (av.) in weight, or approximately bullion 500 fine.

If, therefore, a shipment of bullion 500 fine containing 100 oz. of fine gold were sent to the U.S. Government assay office or mint, the shipper would receive $34.81 per ounce as follows:

100 oz. fine gold at $35 per oz		$3,500.00 gross
Deductions		
Melting	$1.00	
Treatment	2.00 (av. bullion)	
Handling	8.75	11.75
		$3,488.25
Express and insurance		6.97
		$3,481.28

or $34.81 per ounce.

Producing Gold Bullion at Mine versus Shipping a Concentrate

Now, suppose this gold had been recovered in the form of a concentrate obtained by flotation, or gravity concentration, and these concentrates had to be hauled by truck 10 miles to the railroad at a point 200 miles from the smelter and under average conditions as to freight, treatment charges, etc., and assuming the concentrate contained no base metals or silver in sufficient quantity to warrant the smelter paying for them, and that they could be shipped to a copper smelter charging no penalty for the percentage of insoluble, we have the following approximate calculations:

(*a*) 50 tons of concentrates carrying 2 oz. of gold per ton. Smelter payment for 100 oz. gold at 91 per cent of $35.00 = $3,185.00

Deductions			
Hauling 50 tons	@ $ 2.00	= $100.00	
Freight 50 tons	@ 5.00	250.00	
Freight and hauling on 8 per cent moisture		28.00	
Treatment charges	4.00	200.00	
Assaying		4.00	
		$582.00	582.00
			$2,603.00

or $26.03 per ounce.

(*b*) 20 tons of concentrates carrying 5 oz. of gold per ton. Smelter payment for 100 oz. gold at 92.5 per cent of $35.00 = $3,237.50

Deductions			
Hauling 20 tons	@ $ 2.00	= $ 40.00	
Freight 20 tons	@ 10.00	200.00	
Freight and hauling on 8 per cent moisture		19.20	
Treatment charges	4.00	80.00	
Assaying		4.00	
		$343.20	343.20
			$2,894.30

or $28.94 per ounce.

(*c*) 20 tons of concentrates carrying 10 oz. of gold per ton. Smelter payment for 200 oz. gold @ 94 per cent of $35.00 = $6,580.00

Deductions			
Hauling 20 tons	@ $ 2.00	= $ 40 00	
Freight 20 tons	@ 15.00	300.00	
Freight and hauling on 8 per cent moisture		27.20	
Treatment charges	@ 4.00	80.00	
Assaying		4.00	
		$451.20	451.20
			$6,128.80

or $30.64 per ounce.

The difference in the percentage of $35 per ounce paid being set by the smelters according to the gold contents per ton of the concentrates.

COMPARISON

Bullion to U.S. assay office or mint = $34.81 per oz. of gold
(*a*) 2 oz. gold concentrate to smelter = 26.03 per oz. of gold
(*b*) 5 oz. gold concentrate to smelter = 28.94 per oz. of gold
(*c*) 10 oz. gold concentrate to smelter = 30.64 per oz. of gold

If less than carload shipments are made to the smelter the freight rate would be 30 per cent higher and the concentrates must be sacked, otherwise the rate would increase 100 per cent. Truck-haul instead of shipping by railroad would cost approximately $7.50 per ton for 200 miles over good roads irrespective of the value per ton. The concentrates must be sacked or shipped in some type of a container; 50-gal. oil drums are sometimes used, the empties being returned to the mine on the back haul.

These figures are not supposed to be exactly accurate, but to illustrate the general average, under existing conditions, of the benefit of producing gold bullion at the mine as against shipping a concentrate containing the gold to a smelter. There are other factors to be considered, such as: silver and copper contained in the concentrate in sufficient quantity to receive payment from the smelter, and which could be recovered with the gold; the percentage of recovery obtained by concentration or flotation, as against a method of treatment for recovering the free gold only. The figures, however, show conclusively that, other things being equal, it pays the miner to recover at the mine as much of his gold as he can, in the form of bullion and sell it as such to the Government.

Preliminary Investigations

In his enthusiasm over the new price for gold, the miner is apt to overlook fundamentals and forget that the same careful investigation must be made of his mine, its future possibility, local conditions, method for recovering the gold, and the various other factors: determining whether or not it is a legitimate enterprise, as was necessary under old conditions; that the only factor that has changed is an increase of approximately 70 per cent in the market value of his product, a very high appreciation, certainly, which in many cases may change what previously was waste into commercial ore, but which will not obviate the necessity of sufficiently developing the mine; deciding how the ore should be treated; determining what recovery can be obtained; obtaining adequate water supply; estimating the net returns per ton of ore, etc.; and balancing these factors against his total expenses per ton, not forgetting to include a fair amount for retiring the entire investment, and as small gold mines are proverbially mines of comparatively short life, figuring liberally the item for amortization and an amount for working capital sufficient to provide funds until the property is on a self-supporting basis.

Having developed the mine to the point where a reasonably accurate estimate can be made as to the grade of ore, tonnage, daily production, water supply, etc., the next step is to find out whether the ore has commercial values under local conditions and how and what to do to commercialize it. This, in crude terms, means how to convert gold in the ore to dollars in the bank and have sufficient money left over after paying back the investment and all expenses to leave a profit for the investors.

High-grade direct shipping gold ore is not being considered. It requires no discussion; is comparatively unusual; inclined to be illusive and somewhat like manna from heaven. Assuming, therefore, that the ore must be milled, the questions to be decided are: How large a mill and what kind of a mill to build.

Fundamentally, it may be accepted that a mine should always crowd a mill for ore and that a mill should never crowd the mine for feed. Therefore, in deciding the size (daily tonnage) of the mill, it is far better to start with a mill too small than too large, provision being made in laying it out for building additional units at a minimum cost when conditions warrant.

We are considering in this article small mills from 15 to 25 tons per day up to as large as 100 or 150 tons per day; the 15 to 25-ton mills being as small as can be considered commercially practicable—mills that under fairly favorable conditions would cost from $6500 for a 15-ton daily capacity to $45,000 for 100-ton capacity, with an additional $20,000 for the larger mill if flotation and cyanidation of the concentrates have to be considered.

What kind of a mill? This cannot be guessed at with any reasonable expectation that the guess is going to be correct. There are several reliable and established firms that have specialized on testing ores, deciding the correct method of treatment, the proper flow sheet and the necessary equipment to carry out the flow sheet. The most likely treatment for gold ores would come within the following:

1. Recovery of the free gold as amalgam, retorting and shipping bullion.
2. Following the recovery of the free gold as above, flotation and the shipping of the concentrates to a smelter.
3. Same as 2, except that the concentrates are treated by cyanidation, the product is refined and shipped as bullion.
4. Straight flotation and shipping the flotation concentrate.
5. Same as 4 except that the concentrates are treated by cyanidation.
6. Cyanidation. Recovery of the free gold as bullion, followed by cyanidation.
7. Gravity concentration.

It has been shown that gold shipped as bullion brings a higher return per ounce than gold shipped in concentrates to a smelter, because not only does it demand a higher price per ounce but shipping and treatment charges are avoided; therefore every effort should be made to recover as much of the gold as is possible in the form of bullion before subjecting the mill feed to a secondary treatment for additional gold recovery and the production of concentrates to be shipped to a smelter.

Is it advisable to start out with a small makeshift mill with the idea of saving as much of the free gold values as possible, saving the tailings for future re-treatment, and so make the mine furnish some of the capital for development and a permanent plant? This method is sometimes practical and advisable.

Is it better to wait until sufficient ore is developed, then build a simple mill, saving the tailings and, as funds are accumulated, add gravity concentration, or flotation, or cyanidation?

When larger tonnages are indicated (100 to 150 tons per day production) the logical plan is to develop sufficient ore in the mine of high enough value so that the milling of this grade of ore will make a sufficient return at least to pay back the original investment, then go ahead, after having carefully determined the proper method of treatment, and build the full-size mill. It is always more expensive to recover tailings for re-treatment than to treat current feed, and generally better over-all recoveries are obtained.

The necessity for having careful tests made by competent metallurgists on an average sample of the ore, before considering the design of the mill, is of such importance that it may be well to outline the general procedure in carrying out such work.

Sampling

Of prime importance is the securing of a sample from the mine that will be as representative as possible of ore that will be treated in the mill, both as to its physical characteristics and valuable contents.

A good practice to follow, and one that shows foresight on the part of the miner, is to take a few pounds from each mine car or bucket taken from the mine during development work, when working on the ore, and carefully store this, preferably in sacks. Such a sample, which should be an approximate average of fine and coarse material, can later be used as one of the samples for testing purposes. When the mine has been sufficiently developed to warrant the consideration of a mill, the ore in place from the various faces should also be carefully sampled by a competent and experienced engineer. Each face of ore should be cleaned off, short holes drilled and the full width of the ore face blasted down. The sample so obtained from each face of ore should be brought to the surface. If any of the faces of ore are shown to contain ore of markedly different nature from the general average, this ore should be kept separate, otherwise the different samples from the various faces can be mixed and considered one sample.

The larger pieces in the sample should be broken down to about one inch, the entire sample mixed thoroughly by shoveling over several times, coned, spread out and quartered down, remixed and requartered until the necessary sample has been secured, weighing from 500 to 1000 lb. The rejects should be sacked, tagged and stored for future reference. The test sample should be sacked in clean strong sacks (or double sacked); each sack should be tagged inside and outside and shipped to the testing plant.

If a sufficient quantity of mill ore to warrant it has accumulated on the dump, a separate sample might also be taken from the dump.

Information should be furnished with the test sample concerning all the important factors connected with the mill, such as available water supply and its source, whether from the mine or flowing water; the estimated expense of hauling concentrates to the railroad point and the freight rate or truck haul to the smelter; type of power, whether electric, diesel engine or steam and if possible, cost of such power; description of the mill site and how close to the mine the mill can be placed; tailings disposal and whether such tailings will have to be stacked or not. If mine water is to be used for milling purposes, a good sized sample (5 gal. or more) should be sent with the ore, to make sure that it is suitable.

Upon receipt of the samples at the testing plant, a record is made of the shipment, including the name of the owners, location of mine, weight of sample, general nature, etc. A few pieces of the coarser ore are picked out, for microscopic examination, etc., should the test work show that such examination would be of assistance in overcoming any difficulties that may have been encountered in testing. If the shipment consists of one lot, it is then crushed in small crusher and rolls to about 10 mesh, cut down through riffles to about 25 lb. This is ground to 20 or 30 mesh and duplicate head samples cut for assay. If the shipment comprises more than one lot, each lot is handled separately, as described.

A sample from the shipment is panned, and if the panning shows the presence of free gold the assayer is warned to take special precautions in preparing the pulp for assay, examining the fine screen for metallics, etc. All samples are assayed in duplicate, and a duplicate sample is filed for reference. The head sample is usually assayed for gold, silver and further analyzed for any base minerals of which the testing metallurgist deems it desirable to have knowledge, as an aid in conducting the test work.

TESTS

After having obtained the analysis of the ore, the metallurgist lays out a series of small-scale tests using the process, which he believes is most likely to give the best results. The first thing to decide is the probable mesh to which the ore will have to be ground to free the mineral from its gangue. To assist in this decision, an assay screen analysis of an average sample is made and the microscope is used to examine the various mesh sizes. Of late years the metallugist relies on the microscope much more than he used to. Careful microscopic and microphotographic work sometimes discloses the fact that the inclusion between the gangue and mineral is so intimate that no practical grinding will liberate the mineral. Such a disclosure by the microscope will save much time and expense in testing, as it proves that adequate results cannot be obtained by gravity separation or flotation, and the only hope lies in a chemical method of dissolution. The microscope is valuable also in identifying the various minerals carried by the ore.

Tests are made on average portions of the sample by the various processes that suggest themselves, and by a combination of two or more processes, such as amalgamation, flotation, gravity concentration, cyanidation, etc. The tests probably will have to be made at various grindings, combinations of flotation reagents, strength of cyanide solutions, dilutions, time of contact, etc.

Careful records of all tests are kept, with notes of any important or interesting facts that have been disclosed. The results from each test are worked out to determine the percentage of recovery at the various steps of the different treatments, ratio of concentration, grade of concentrates and final tailing loss. These results are carefully compared with each other, not only the results themselves, but also the method of treatment used to obtain them. The results are carefully considered in conjunction with the location of the mine, local conditions, water and power, size of the contemplated mill, as these factors have a bearing second only to the metallurgical results. It is manifest that there is no good in deciding, in the testing, on a method of milling that could not, at reasonable expense, be applied at the mine.

Following this analysis of results on the first series of tests, additional test work is usually carried out on larger samples, using the method of treatment that gave most promise in the smaller tests, and finally a flow sheet is decided upon. The next step is to convert the saving obtained into returns per ton of crude ore milled. This is simple if the values recovered in the mill are entirely represented by gold bullion. If, however, all or part of the mill savings are represented by a concentrate that has to be shipped to a smelter, additional calculations are required. To enable this calculation to be made correctly, a smelting schedule under which the concentrates would be purchased must be obtained, freight rates from the nearest railroad point to the smelter and hauling charges from the mine to the railroad point. An average smelting schedule and its application to concentrates obtained in testing are shown in Table 1.

TABLE 1.—*Smelting Schedule and Its Application to Concentrates*

AVERAGE SMELTING SCHEDULE

Gold.—If 0.03 oz. per ton or over, pay for all at $19 per ounce, nothing paid if less than 0.03 oz. per ton. As a preliminary settlement, adjusted settlement based on the market price of gold.

Silver.—Pay for 95 per cent of contents (minimum deduction 1 oz.) at 64⅛¢ per ounce, less 1½¢ per ounce.

Table 1.—(*Continued*)

Lead.—Deduct from wet lead assay 1.5 units and pay for 90 per cent of the balance at the average daily published quotations for the calendar week less 1½¢ per pound of lead accounted for. Nothing paid for lead if less than 5 per cent wet assay.

Copper.—Deduct from the wet copper assay 1.3 units and pay for 100 per cent of the remainder at daily net refinery quotations as published in the *Engineering and Mining Journal*, for the calendar week, preceding the date of arrival at the plant, less a deduction at 6¢ per pound. (This refers only to copper contents of a lead-copper concentrate shipped to a lead smelter.)

Iron.—All credited at 6¢ per unit.

Deductions

Base Charge.—Charge is $2 per net dry ton for 30 per cent settlement lead; add 10¢ per ton per unit of lead under 30 per cent; credit 10¢ per ton per unit over 30 per cent.

Insoluble.—All charged at 10¢ per unit.

Zinc.—5 per cent free; charge for excess at 30¢ per unit.

Sulfur.—2 per cent free; charge for excess at 25¢ per unit. Maximum $2.50 per ton.

Lead-copper Concentrate Assaying

Gold	1.383 oz.	Iron	18.62 per cent
Silver	44.67 oz.	Zinc	6.88 per cent
Lead	23.88 per cent	Insoluble	8.42 per cent
Copper	14.74 per cent		

Ratio of concentration—15.70 to 1 (6.37 tons from 100 tons of ore)

Assumed Metal Prices

Gold	$19.00 per oz.	Lead	4.00¢ per pound
Silver	64⅛¢ per oz.	Copper	8.00¢ per pound

Value per Ton of Concentrate

Gold	1.383 oz.	$19.00	per oz. =	$26.28
Final settlement of gold				17.73
Silver	44.67 oz.	@ 64⅛	cents per oz.	26.58
Lead	23.88 per cent	@ 4	cents per lb.	10.07
Copper	14.74 per cent	@ 8	cents per lb.	5.38
Iron	18.62 per cent	@ 6	cents per unit	1.12
				87.16 gross value per ton

Gross value per ton		$87.16
Deductions		
Treatment charge	$2.76	
Insoluble	0.84	
Sulfur (maximum chg.)	2.50	
Zinc 6.88 per cent	.56	6.66
		$80.50
Freight + 10 per cent moisture	3.30	
Hauling	2.00	5.30
		$75.20 net value per ton

$$\frac{6.37 \text{ tons per 100 tons ore} \times 75.20}{100} = \frac{\$479.02}{100} = \$4.79 \text{ net returns per ton ore.}$$

This shows that concentrates of the analysis given, sold under the smelting schedule shown, after deducting all charges and penalties imposed would return \$4.79 per ton of ore milled—not deducting cost of milling. From the net returns per ton of crude ore, as represented by the net returns for bullion shipped to the mint or Government assay office, plus the net returns per ton of ore received from the smelter for concentrates, must be deducted the cost of mining, development work, milling, taxes and insurance, administration expenses and an amount per ton to retire the entire investment cost in a reasonable term of years, depending upon the expected life of the mine.

There is no other way to determine the net value of the ore that has to be treated in a mill than by carefully conducted tests on the ore by metallurgists experienced in such work, who are able properly to interpret the test results into practice, not only as to metallurgy but also as to the equipment most advisable to correctly carry out the indicated flow sheet. Having selected the flow sheet and the right equipment, the correct arrangement of such machinery and the design of the plant itself should be intrusted to competent mill-designing engineers.

Water, Power and Transportation

An adequate water supply is almost as essential as an adequate ore supply—next to being short of ore, to have an unreliable or insufficient water supply is the greatest handicap in wet methods of ore treatment. A plant for the recovery of the free gold only should have available a supply of at least 6 gal. of water per minute for every 10 tons of ore to be milled per 24 hr., and for safety 7 gal. per minute would be better. This should be enough also, with reasonable care, for a flotation addition to the plant. If the final treatment of the entire mill feed is to be by cyanidation, and the final tailings thickened, less water would be required —2½ gal. per minute would be sufficient to treat 10 tons per 24 hr., and 1 gal. per minute if the cyanide tails are filtered after thickening. It is generally possible to recover about 60 per cent of the water in the mill tailings for re-use in the mill, either by dams or some standard thickening equipment whereby the tailings are automatically thickened and part of the water recovered, clear enough to be used in the mill. In some localities a separate water supply must be secured for domestic purposes, for boilers if steam power is used and for cooling water if gas or diesel engines are to be the source of power.

Next in importance to the ore and water supply would be the power supply. This may be any of the following according to the location of the mine: (1) electric power purchased from a public utility company, (2) electric power generated at the property, (3) water power, (4) diesel or semi-diesel engines, (5) gasoline engines, (6) steam.

If there is a public power line within reasonable distance of the mine, it is generally conceded that purchased power is the most satisfactory. It permits the use of individual motors, lowers the first cost of the plant and with present-day efficiency is subject to few power interruptions.

To generate hydroelectric power at the mine calls for a good and constant head of water and a rather high initial investment, but if the water is available and the plant properly designed and installed it will furnish very cheap power. Usually, however, the investment is not warranted by a small gold mine.

Water power by means of a Pelton wheel or turbine means very cheap power, but must be carefully investigated and all the factors studied, especially winter conditions in the colder districts and summer flow in hot districts.

Diesel or semi-diesel engines are popular where electric power is not available and where the fuel haul is not too long or expensive. They furnish power at a reasonable cost, but call for a steady employment of an experienced operator, and cost from $80 to $100 per horsepower installed.

Gasoline engines are adaptable only for the smaller mills, up to, say, 25 tons per day. They are reasonably reliable and efficient.

Steam power, especially in the outlying districts of the United States, is not much used. Wood as a fuel is generally scarce and it is difficult to keep a good supply of fairly dry fuel on hand. As a general rule, coal is too costly delivered at the mine. The diesel engine, to a large extent, has taken the place of the steam engine as a source of power for mills where electric power is not available.

The horsepower per ton required for small gold mills is necessarily a variable quantity and controlled by the daily tonnage treated, the final grinding necessary, the flow sheet, etc. Generally speaking, it will vary from a low of 1½ hp. to a high of 3 hp. per daily ton. If internal combustion engines are used as a source of power, special care must be taken not to overload them above their rated working capacity and to provide the correct type of fuel and cooling water.

An item often overlooked, or not adequately figured in estimating the capital required for developing a mine and equipping it to the production stage, is the method of transportation to and from the mine. Many small gold mines are in isolated districts, some distance from the railroad or highway. The development of truck hauling has materially simplified their problems. Plants producing bullion only require little up or down freight to and from the railroad, but it is essential that road connections be made to the nearest highway (neglecting the few properties that can be served by airplane only, and those that rely on barges and lakes for their transportation, as in some localities in Canada). The building and upkeep of the necessary private road to the mine should be carefully investigated and the cost provided for. No general estimate of cost can be placed on this, the many variables making it impossible.

Equipment

Having developed the mine sufficiently to warrant the erection of a mill and having had the ore tested to determine the correct method of treatment and the recoverable values, the next question to decide is the type of equipment required. This calls for careful consideration and expert advice in fitting the equipment not only to the mill immediately under consideration, but with a view to future extension.

Some of the new devices that have come on the market for recovery of free gold, supposed to be especially adapted for the smaller properties producing a few tons per day, have merit. The chief criticism might be that usually they are too lightly constructed for the every-day wear and tear and severe duty they are called upon to withstand, and that in the endeavor to make them light and compact they have also been made somewhat complicated, with the various renewable parts inaccessible. Some of these devices serve a useful part during development in determining the value of the ore. Generally, however, the cost of such refinement is too heavy for small mines.

The increased price for gold, having stimulated gold mining, has resulted in more attention being paid to methods for recovery of free gold and several useful innovations have been introduced into gold mills during the last two or three years, among which may be mentioned the following: (1) ball-mill grinding as a substitute for stamps; (2) hydraulic traps or jigs for recovery of coarser gold, instead of stationary amalgamating plates; (3) blanket strakes of various types for recovering the fine free gold, in place of stationary or shaking amalgamating plates; (4) a revival of the clean-up barrel or grinding pan for the gold caught in the traps or on the blankets.

The use of ball mills in closed circuit with classifiers for grinding, instead of the old-fashioned stamps, is probably responsible for most of the changes. The stamp seems to have become ancient history. One wonders if the present-day operator does not sometimes look with rather envious eyes on the days when the stamp mill, with its simplicity, comparative ease of operation, even distribution of discharge, ability to handle coarse feed with ease, etc., was the fashion. One of the drawbacks of the ball mill and classifier is the tendency to hold back free gold in the grinding circuit. Often it is found that there is more gold held up in these machines than is recovered from the classifier overflow. This has been largely overcome by the introduction of hydraulic traps, or jigs, on the ball-mill discharge, in the circuit between the mill and classifier.

The hydraulic traps are very simple affairs, as illustrated in Fig. 1; sometimes two or three are used in series. The bottom of the cone is fitted with a valve for clean-up, which can be locked against theft. It is surprising how much gold is recovered by this means. In one plant in Canada, as much as 88 per cent of the gold in the mill feed is saved in the hydraulic traps.

Blanket strakes, one of the oldest methods for recovering free gold, are now again being used extensively, but whereas previously they generally supplemented amalgamation plates they now often replace them.

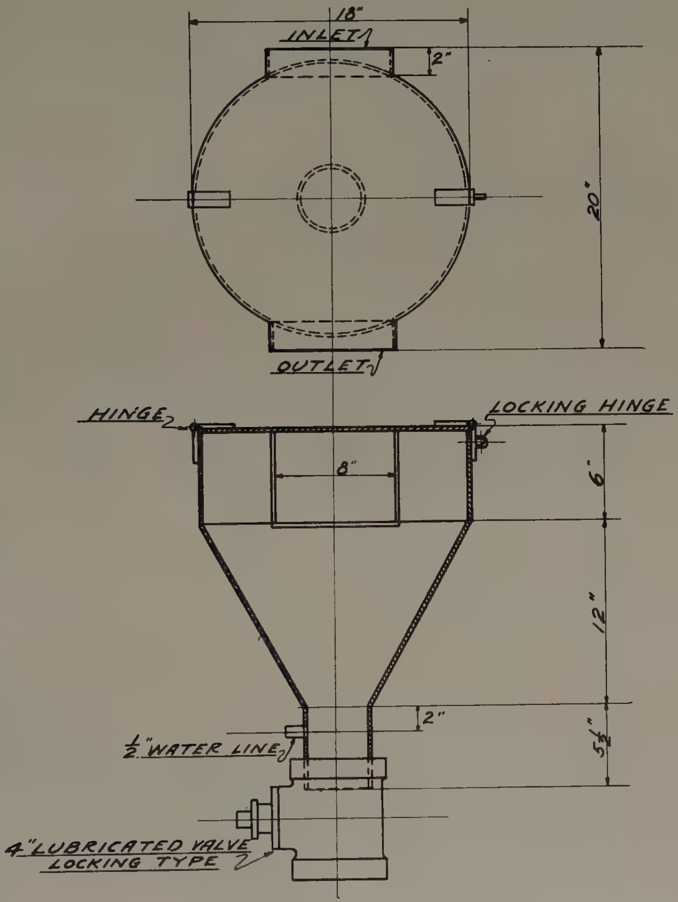


Fig. 1.—Hydraulic gold trap.

Various different types of materials are used for the blanket, the most popular being English pulp-sifting cloth, manufactured by James Johnson of Manchester, England, and stocked by various agencies in the United States and Canada. It retails in the United States at $3.50 per lineal yard, 36 in. wide. It is corduroy material with ribs of the material running lengthwise, and comes in rolls 77 yd. long by 28 and 36 in. wide.

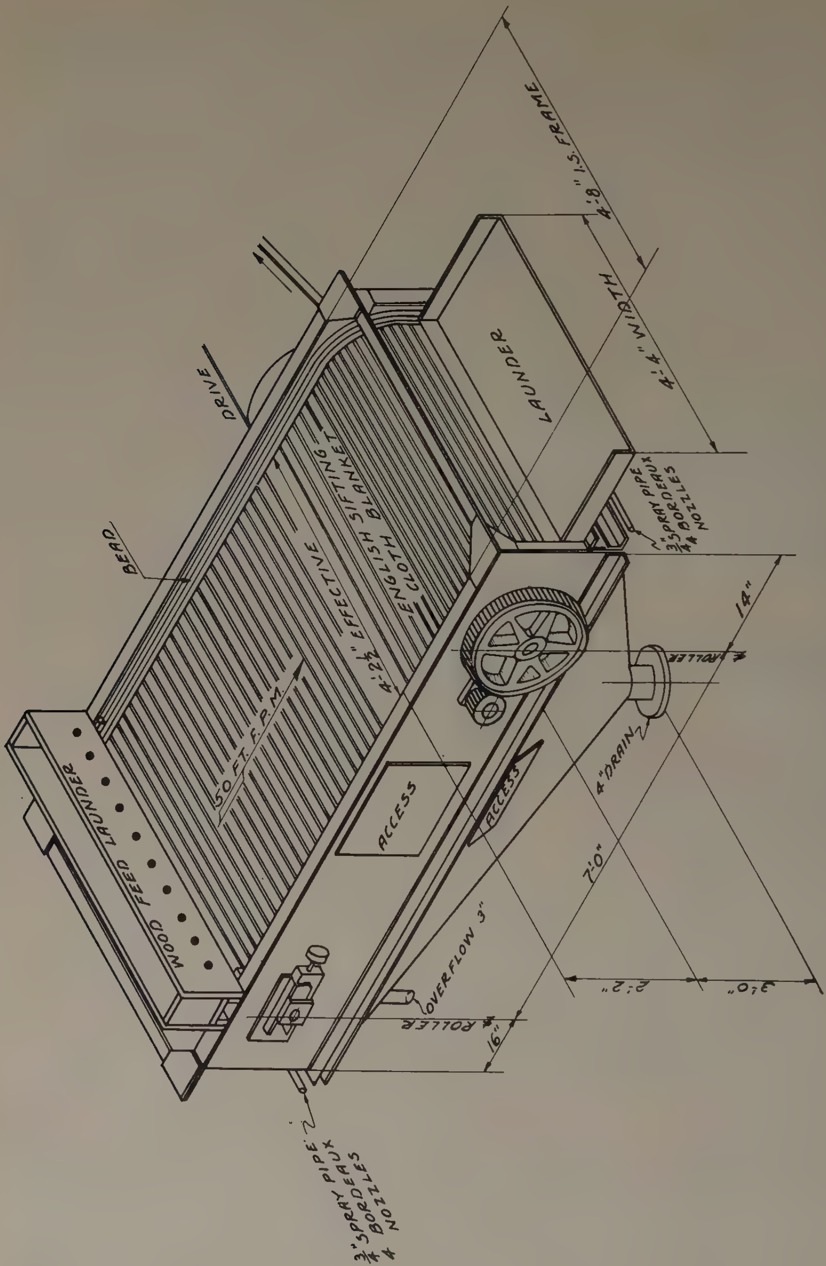


FIG. 2.—SELF-WASHING BLANKET TABLE.

The corduroy strakes are generally 4 ft. wide by 5 to 6 ft. long, laid on a specially prepared table set at a pitch of 1 to 8, the ribs of the corduroy being at right angles to the flow of the pulp.

The pulp feed to the corduroy strakes is generally diluted to about 4 water to 1 solids and can be comparatively coarse (minus 16 mesh). At the Lake View and Star mines of Western Australia, the strakes have a capacity of 3½ long tons per square foot of area. At the Dome mines, Canada, 28 blanket tables, each 27 sq. ft., take care of a primary load of 1500 tons plus a circulating load of 2000 tons.

The method of operating corduroy blankets consists of periodically removing them from the tables for washing and replacing them with washed strips. This is done from three to five times per 8-hr. shift. When the blankets are removed, they are folded and rolled with the product on the inside and taken to the washing tank. The collected concentrate is washed off vigorously. The concentrate collected in the tank, to which is sometimes added the gold saved in the hydraulic traps, is delivered to a clean-up barrel 3 to 4 ft. in diameter by 5 ft. long with a charge of about 500 lb. of 1 to 1½ in. balls. The concentrate is ground in the barrel for a few hours, then mercury is added; the barrel is revolved for an hour, then dumped; the balls are screened out and the product and mercury are sent to a clean-up pan. The solids are washed out, the thin amalgam is drained off; the amalgam is cleaned with hot water, the iron removed with a magnet and the amalgam pressed and retorted.

Some trouble is caused in the use of blanket strakes by the amount of fine iron from the ball mill that collects in the ribs of the corduroy together with the valuable material from the ore. A high grade of forged ball should be used to minimize this. On large installations, a small magnetic separator may be used to remove the iron before the concentrates are sent to the grinding barrel.

The cost of labor involved in washing and changing blankets is from 3 to 5¢ per ton, depending on the size of the plant. In some cases a continuously operating, self-washing table, somewhat along the lines of an old-fashioned vanner without the side motion, has been designed to eliminate most of this labor cost (Fig. 2).

Milling at Montezuma-Apex Mining Company

The following information was furnished through the courtesy of Mr. J. A. Norden, Manager of the Montezuma-Apex Mining Co., and is published by permission of Mr. R. F. Haffenreffer, President:

Fig. 3 is a flow sheet illustrating the milling procedure at the plant of the Montezuma-Apex Mining Company, a subsidiary of the Utah-Apex Mining Company, located at Nashville, California. This plant went into operation in the early part of 1933 and most of the principal equipment was transferred from the Utah-Apex mill at Bingham Canyon, Utah, which, owing to the low prices of lead, copper and zinc, was closed down in 1933.

The Montezuma ore values are in the form of metallic gold and gold associated with sulfides of iron, together with small amounts of antimony, bismuth, lead and copper. The gangue material is quartz, graphitic slates and carbonaceous shale. The free gold values range in size from particles of pinhead size to very fine dust.

Grinding is done in two Marcy ball mills (size 64½), the capacity of each unit being approximately 130 tons per day in reducing a rather coarse feed to flotation size (approximately 65 mesh).

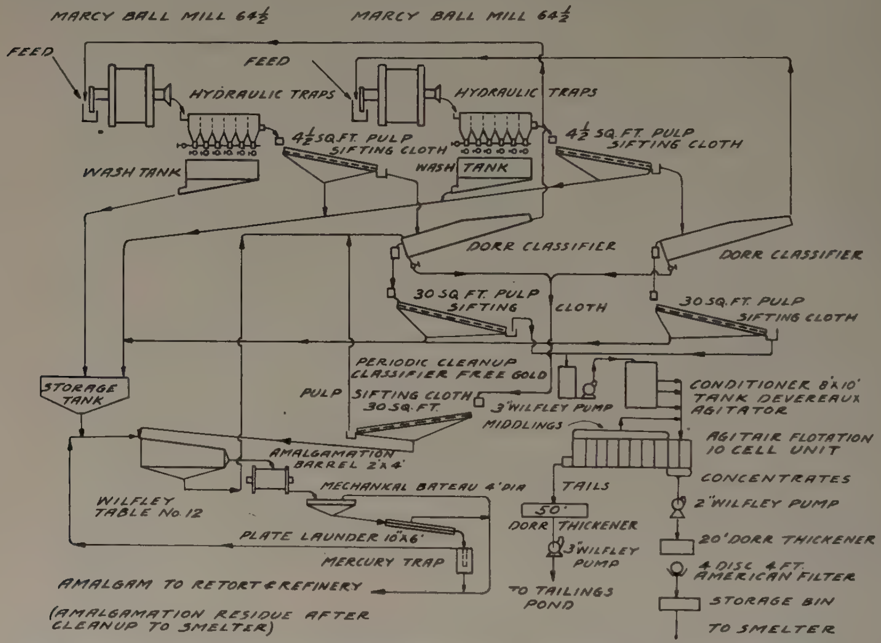

MARCY BALL MILL 64½
MARCY BALL MILL 64½
FEED
FEED
HYDRAULIC TRAPS
HYDRAULIC TRAPS
4½ SQ. FT. PULP SIFTING CLOTH
4½ SQ. FT. PULP SIFTING CLOTH
WASH TANK
WASH TANK
DORR CLASSIFIER
DORR CLASSIFIER
30 SQ. FT. PULP SIFTING CLOTH
30 SQ. FT. PULP SIFTING CLOTH
STORAGE TANK
PERIODIC CLEANUP CLASSIFIER FREE GOLD
PULP SIFTING CLOTH 30 SQ. FT.
3" WILFLEY PUMP
MIDDLINGS
CONDITIONER 8'x10' TANK DEVEREAUX AGITATOR
AGITAIR FLOTATION 10 CELL UNIT
AMALGAMATION BARREL 2'x4'
MECHANICAL BATEAU 4' DIA
WILFLEY TABLE No. 12
TAILS
CONCENTRATES
50' DORR THICKENER
2" WILFLEY PUMP
PLATE LAUNDER 10"x6'
3" WILFLEY PUMP
20' DORR THICKENER
MERCURY TRAP
4 DISC 4 FT. AMERICAN FILTER
AMALGAM TO RETORT & REFINERY
TO TAILINGS POND
STORAGE BIN
(AMALGAMATION RESIDUE AFTER CLEANUP TO SMELTER)
TO SMELTER

FIG. 3.—FLOW SHEET OF MILL AT MONTEZUMA-APEX MINING CO. (*Courtesy Mr. J. A. Norden, Manager.*)

The pulp from the ball mills is passed through hydraulic traps and then over short sections of pulp-sifting cloth before entering Dorr classifiers, which are operated in closed circuit with the mills. The classifier overflow passes over stationary tables covered with pulp-sifting cloth, after which it is pumped to a conditioning tank before flotation.

Approximately 75 per cent of the values recovered is in the form of metallic gold, which is taken from the traps and the pulp-sifting cloth in a concentrated form and then amalgamated in the old-fashioned amalgamation barrel. The cloth tables are cleaned once each 8-hr. shift. The traps are bled daily and thoroughly cleaned twice monthly. This product is placed in storage and further concentrated periodically on a small Wilfley table, the concentrate from which is amalgamated, and the tailing is returned to the main mill circuit through the classifiers. The classifiers are thoroughly cleaned out once monthly, the sands being passed over pulp-sifting cloth and then returned to the mill circuit.

In the amalgamating process, which is a bi-weekly operation, the accumulated gold concentrate is first ground for several hours to scour the mineral particles, thus increasing the amenability of the gold to amalgamation. Excess mercury is then added and the barrel is continued in operation for an additional 2 to 3 hr., after which it is discharged into a mechanical batea. The overflow from the batea passes through a short section of launder in which are placed amalgamation plates. This launder discharges into a mercury trap and the overflow from this is passed over a Wilfley table, the purpose of which is to recover any amalgam and free mercury that may have passed over the batea, plates and trap, and which is readily segregated at the upper edge of the table and thus separated from the remainder of the residue which is shipped as a special product. In this procedure every possible care is used to avoid returning any mercury to the main mill circuit, because in passing through pumps, or in the violent action of the ball mill, or in flotation machines the mercury will be floured and when in this condition is apt to attach itself to gangue particles and carry out gold values into the tailing.

In the flotation circuit the pulp is conditioned for about 15 min. with sodium sulfide, copper sulfate, Aerofloat No. 25, amyl xanthate and cresylic acid. The concentrate is taken from the first two units of a 10-cell machine. A middling product from the last eight cells is returned to the head of the circuit. The concentrate is thickened, filtered and shipped as a smelter product.

In the design and development of the milling process at this property the prime objective, of course, is a maximum recovery and a reduction of as much as possible of the ore values into bullion form from which the greatest monetary return per ounce of gold may be realized. So far as the readily recovered free gold values are concerned, this seems to have been achieved in a simple and effective manner, but the monetary recovery from gold in the form of concentrate cannot be considered as entirely satisfactory. The graphite and carbon contained in the ore floats as readily as the sulfides and carries with it an undue amount of insoluble material, which results in a siliceous, comparatively low-grade concentrate and which, on account of its carbon content, is not readily amenable to satisfactory cyanide treatment. Any attempt to depress the carbon results in high tailing losses.

The mill is operated on a three-shift basis with one operator per shift. The primary crushing plant is operated on a two-shift basis with one operator per shift. In addition, there is a mill superintendent and one repairman. On clean-up days or when major repairs are required other help is pressed into service.

Milling results during a typical period are as follows:

		Per Cent
Gold per ton in concentrate recovered, oz	0.0500	
Gold per ton in bullion recovered, oz	0.1312	
Total recovered, per cent	0.1812	92.69
Tailing loss by assay, per cent	0.0114	5.68
In mill circuit or unaccounted for, per cent	0.0032	1.63
Gold per ton in mill feed by assay, oz	0.1948	100.00

While experience in this plant does not cover a sufficiently long period of time to determine cost definitely, the results thus far indicate that on the basis of 3000 tons per month the operating cost is approximately $1 per ton. While on the basis of 5000 tons per month, which is now being milled, the cost is approximately $0.80 per ton. A considerable part of this cost is applicable to fine grinding and there is an opportunity for a saving here inasmuch as only a small Blake-type machine is being used in the primary crushing plant. The introduction of a secondary crusher and the reduction of the ball-mill feed to a minus inch product will probably increase the mill capacity 20 per cent and at the same time reduce the grinding cost materially.

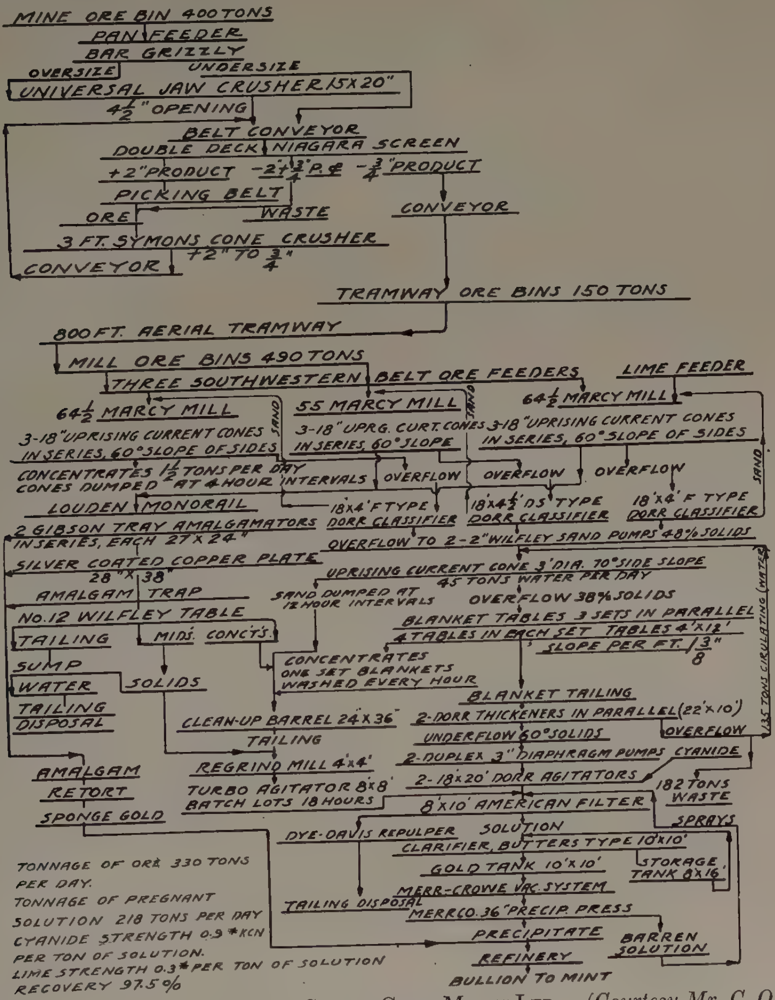
Siscoe Gold Mines Limited

Through the courtesy of Mr. C. O. Stee, Mine Manager, Siscoe Gold Mines Limited, Siscoe, Quebec, Canada, I have been furnished, and permitted to publish, the present flow sheet of the mill (Fig. 4) which is now treating 330 tons per day. The flow sheet also shows the new crushing plant under course of construction. The following information is taken from Mr. Stee's letter:

An interesting feature of this flow sheet is the type of tramway employed; namely, the shuttle type, with two parallel ropes as carriers and a 2-ton bucket which rides on four wheels, is automatically self-dumping at the mill end, the whole unit, including the loading of the bucket, being handled by one man. The capacity is 40 tons per hour.

The concentrating cones at the discharge end of the ball mills, from which a recovery of approximately 88 per cent of the free gold is made, are particularly interesting. This concentrate, which does not exceed 2 tons in 24 hr., is transported from the cones by a Louden monorail to the bin above the Gibson amalgamators. The present amalgamating unit is entirely enclosed by heavy wire screening with a tight ceiling above and a concrete floor below, the concrete extending up from the floor all around a minimum distance of 8 in. In this floor sumps are provided which are periodically cleaned and to which all spillage is sluiced. One man conducts the amalgamating work within this enclosure, including the retorting of the amalgam. When this work is completed, he can ring a bell in the superintendent's office and an escort is provided when the gold is removed for weighing and placing in the safe.

This mill has perhaps a greater area of blanket tables, per ton passing over, than is found in other mills. This was arrived at through a series of experiments over a period of time. The area that should be installed for any class of ore is largely a matter of experimentation. When blankets are removed from the tables for washing, they are folded and transported in tight-bottomed cement buggies to prevent loss of free gold. From the washing tank the concentrate is sluiced to a clean-up barrel where the gold is amalgamated. This is also included within the screening mentioned above.


MINE ORE BIN 400 TONS
PAN FEEDER
BAR GRIZZLY
OVERSIZE
UNDERSIZE
UNIVERSAL JAW CRUSHER 15x20"
4½" OPENING
BELT CONVEYOR
DOUBLE DECK NIAGARA SCREEN
+2" PRODUCT
-2+¾" P.&
-¾" PRODUCT
PICKING BELT
ORE
WASTE
CONVEYOR
3 FT. SYMONS CONE CRUSHER
+2" TO ¾"
CONVEYOR
TRAMWAY ORE BINS 150 TONS
800 FT. AERIAL TRAMWAY
MILL ORE BINS 490 TONS
THREE SOUTHWESTERN BELT ORE FEEDERS
LIME FEEDER
64½ MARCY MILL
55 MARCY MILL
64½ MARCY MILL
SAND
SAND
SAND
3-18" UPRISING CURRENT CONES IN SERIES, 60° SLOPE OF SIDES
3-18" UPRG. CURT. CONES IN SERIES, 60° SLOPE
3-18" UPRISING CURRENT CONES IN SERIES, 60° SLOPE OF SIDES
CONCENTRATES 1½ TONS PER DAY
CONES DUMPED AT 4 HOUR INTERVALS
OVERFLOW
OVERFLOW
OVERFLOW
LOUDEN MONORAIL
18'x4' F TYPE DORR CLASSIFIER
18'x4½' DS TYPE DORR CLASSIFIER
18'x4' F TYPE DORR CLASSIFIER
2 GIBSON TRAY AMALGAMATORS IN SERIES, EACH 27 x 24"
OVERFLOW TO 2-2" WILFLEY SAND PUMPS 48% SOLIDS
SILVER COATED COPPER PLATE 28" X 38"
UPRISING CURRENT CONE 3' DIA. 70° SIDE SLOPE
45 TONS WATER PER DAY
AMALGAM TRAP
SAND DUMPED AT 12 HOUR INTERVALS
OVERFLOW 38% SOLIDS
No. 12 WILFLEY TABLE
BLANKET TABLES 3 SETS IN PARALLEL
4 TABLES IN EACH SET TABLES 4'x12'
SLOPE PER FT. 1⅜"
TAILING
MIDS.
CONC'TS.
CONCENTRATES ONE SET BLANKETS WASHED EVERY HOUR
SUMP
WATER
SOLIDS
BLANKET TAILING
TAILING DISPOSAL
CLEAN-UP BARREL 24 x 36"
2-DORR THICKENERS IN PARALLEL (22'x10')
TAILING
UNDERFLOW 60° SOLIDS
OVERFLOW
135 TONS CIRCULATING (WATER)
AMALGAM
REGRIND MILL 4'x4'
2-DUPLEX 3" DIAPHRAGM PUMPS
CYANIDE
RETORT
TURBO AGITATOR 8'x8'
BATCH LOTS 18 HOURS
2-18'x20' DORR AGITATORS
182 TONS WASTE
SPONGE GOLD
8'x10' AMERICAN FILTER
SPRAYS
DYE-DAVIS REPULPER
SOLUTION
CLARIFIER, BUTTERS TYPE 10'x10'
GOLD TANK 10'x10'
STORAGE TANK 8'x16'
MERR-CROWE VAC. SYSTEM
TAILING DISPOSAL
MERRCO. 36" PRECIP. PRESS
PRECIPITATE
BARREN SOLUTION
REFINERY
BULLION TO MINT

TONNAGE OF ORE 330 TONS PER DAY.
TONNAGE OF PREGNANT SOLUTION 218 TONS PER DAY
CYANIDE STRENGTH 0.9 # KCN PER TON OF SOLUTION.
LIME STRENGTH 0.3 # PER TON OF SOLUTION
RECOVERY 97.5%

FIG. 4.—FLOW SHEET OF MILL AT SISCOE GOLD MINES LTD. (*Courtesy Mr. C. O. Stee, Manager.*)

The cyanide unit, which receives the tailings from the blanket tables, has only about 6 per cent of the original values in the ore left to work upon. Its recovery is about 3.5 per cent. From the cyanide unit the mill seems to be short on settling and filtering capacity, but this is being corrected by adding two extra disks to the 8 by 10-ft. American United Filter. After this is put into operation there may also be installed trays in the thickeners, to increase their capacity, if it is found necessary and economical.

Flotation tests, conducted over a year ago, indicate that Siscoe ore, at the present time, cannot be floated economically; furthermore, a very high extraction, with low costs, is being obtained with the process now in use. The total milling costs have averaged, during recent months, $1.10 per ton. A paper on amalgamating and cyaniding on Siscoe island appeared[1] in 1932, but a number of changes have been made since that time.

Recovery of Free Gold

It will be noted that both the Montezuma-Apex and the Siscoe mills use hydraulic traps (concentrating cones) for recovering the free gold in the ball-mill classifier circuit and sifting cloth for the fine gold in addition to stationary or shaking amalgamating plates. Particular note should be made of Mr. Stee's statement that 88 per cent of the free gold in the Siscoe ore is saved by these traps, and Mr. Norden's statement that in the Montezuma-Apex mill similar traps save 75 per cent of the gold in his ore.

In some mills treating free gold ore, instead of hydraulic traps, a unit flotation machine of somewhat special design is installed between the ball mill discharge and the classifier, with efficient results. This, however, is more applicable to larger mills. Another device placed in a similar position and for a similar purpose is a small one or two-compartment Hartz-type jig of special design. I have been advised that this jig is used successfully in the mills at the Bralorne mine, B.C., and the Murchie mine in Grass Valley. In the latter mill it is saving not only coarse but very fine gold with a ratio of concentration of one to nine thousand. It would appear, however, that considering first cost, simplicity and efficiency, the hydraulic trap is the most popular device for the particular purpose desired.

With reference to the recovery of free gold by flotation, Leaver, Woolf and Head say:[2]

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[1] C. O. Stee: Almalgamating and Cyaniding on Siscoe Island, Quebec. *Eng. & Min. Jnl.* (1932) **133**, 319; also in *Profitable Mine & Mill Practice.*

[2] E. S. Leaver, J. A. Woolf and R. E. Head: U.S. Bur. Mines *Rept. of Investigations* 3226 (1934) 15.

Clean metallic gold, minus 60 mesh in size, can be floated successfully from a siliceous gangue or a siliceous gangue containing pyrite with the usual flotation reagents used for sulphide ore.

An almost complete recovery of clean metallic gold can be made by flotation, provided the ore is all ground to 60 mesh and no interferring slime is present in the flotation feed.

Gold coarser than 40 mesh is not recovered in the usual flotation concentrate.

These statements check very closely the conclusions I have reached, but it appears to me that, unless the gold is so tarnished that it cannot be collected by mercury, it is usually more profitable to recover the free gold by methods illustrated in Figs. 3 and 4, using flotation only to recover the gold carried by the pyrite or other base minerals together with as much silver as possible, should the ore carry silver values. If such flotation concentrates can be successfully treated by cyanidation, a small cyanide annex for this purpose should soon pay for itself in the extra price received for bullion as against shipping concentrates to the smelter. A careful analysis should be made to balance costs plus tailings loss against the additional returns.

CONCLUSIONS

The high price of gold coupled with the low price of base metal, has so stimulated gold mining that there hasbeen some tendency to overlook or disregard fundamentals that have always existed and still exist concerning the necessity for proper investigation of the mine, the mill and the various other factors in deciding whether it is a legitimate venture or not. Many small gold-mining ventures have been unsuccessful because of lack of capital to put them on a profitable basis. This often tempts the gold miner to forego the expenditure to pay for expert advice as to the mine, a well laid out development plan, and determining the proper treatment for the ore. This is always costly and often means disaster.

If in the few remarks I have made, I have been able to impress upon the owners of the types of gold mines under discussion that gold mining and milling, like every other business enterprise, call for advice and assistance from those who are trained and competent to give such advice and assistance, I shall have been amply repaid.

Increasing Gold Recovery from Noranda's Milling Ore

By C. G. McLachlan,* Member A.I.M.E.

Two papers dealing with Noranda's milling operations have already been presented. The first[1] of these covered the initial metallurgical problems connected with the treatment of the ore, while the second[2] described the development of the mill from a 500-ton unit in 1928 to one with a daily capacity of 3000 tons less than six years later.

At the time the second paper was written the equipment which had been installed to bring the mill capacity up to 3000 tons was being tuned into production and for this reason, though each step had previously been tested by a large-scale pilot operation, it was thought advisable to withhold metallurgical data until any starting-up difficulties had been overcome and anticipated requirements fulfilled.

These requirements called for an improvement in gold recovery without any lowering of the previous ratio of concentration, and the pages that follow deal with the methods by which this result was obtained.

An idea of the character of Noranda concentrating ore will be gained from an inspection of the following mineralogical analysis of the ore treated by the mill during 1933:

Mineral	Content	Mineral	Content
Chalcopyrite, per cent	6.8	Silica and silicates, per cent	18.3
Pyrite, per cent	22.1	Gold, native and as telluride, oz. per ton	0.15
Pyrrhotite, per cent	51.5	Silver, probably as argentite, oz. per ton	0.35
Magnetite, per cent	0.5		

These figures, which are typical, show that over 73 per cent of the ore treated consists of pyrite and pyrrhotite. Determination of the proportion of unrecovered gold associated with each of these minerals was therefore of primary importance in developing a suitable method for improving gold recovery.

As pyrrhotite is magnetic, this information was obtained by making magnetic sorting tests on samples of the mill tailing and screening the material thus segregated. This showed that the magnetic material finer

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Manuscript received at the office of the Institute Oct. 19, 1934.

* Concentrator Superintendent, Noranda Mines, Limited, Noranda, Quebec.

[1] C. G. McLachlan: Twelve Month's Milling at Noranda. *Trans.* Canadian Inst. Min. and Met. (1930) **33**, 319.

[2] C. G. McLachlan: The Development of Concentrating Operations at Noranda. *Canadian Min. Jnl.* (1934) **55**, 175.

than 200 mesh carried about 0.02 oz. of gold per ton, while the nonmagnetic residue assayed considerably higher. As the nonmagnetic residue consisted mainly of pyrite and silica, further tests were undertaken to determine which of these minerals carried the gold.

This last determination was made by taking samples of the mill tailing and floating both the pyrite and the pyrrhotite away from the silica. The siliceous residue was then amalgamated to guard against the presence of tarnished gold that had failed to float. The results of the completed test showed that the silica carried only a trace of gold, and similar tests made without the amalgamation step did not materially increase the amount of gold found.

As the foregoing results pointed very strongly to the fact that the gold was being carried by the pyrite, an optical sorting test was made on a composite nonmagnetic residue. From this residue the pyrite was sorted grain by grain until a quarter of an assay ton was obtained. Assay of this sorted material confirmed the fact that the pyrite was carrying the gold.

Following this initial work a large number of tests were carried out in the laboratory in which samples of the mill tailing were reground and re-treated as a preliminary step to recovering the gold. These tests, however, failed to produce any worth-while results, and were followed by others in which an attempt was made to segregate the pyrite from the rest of the mill tailing by flotation. The advantages to be gained by such a step were: (1) The concentrate being higher in gold than the mill tailing, the relative effectiveness of different treatments would be more apparent; (2) separation of the pyrite would permit tests being made on a segregated refractory product which, as the chemical behavior of pyrite and pyrrhotite is very different, was particularly important if a cyanide step were to be developed.

The differential separation of pyrite from pyrrhotite was successfully accomplished in the laboratory in the fall of 1930, by floating the former from the latter, and by the end of that year some progress had been made in extracting the gold from the pyrite concentrate thus produced by subjecting it to a further flotation step following fine grinding.

At this stage, however, it was felt that some time ought to be spent in checking over the previous work to be sure that no simpler method could be used to improve gold recovery than was promised by this procedure. During the period that elapsed while this check-up was taking place four separate but contemporaneous investigations were undertaken, each of which was connected with different phases of the problem, as follows:

1. Under the directorship of Dr. F. M. G. Johnson, Dean of Science at McGill University, Dr. Maass and Dr. Steacie, of the Faculty of Chemistry, developed a method by which the collecting properties of

different reagents could be measured quantitatively under conditions similar to those existing in the mill flotation circuit. The results of this investigation are now in course of publication and therefore will not be described here. The method developed furnished us with valuable information on the floatability of gold and gold telluride, which it would have been very difficult to obtain so quickly or conclusively by regular laboratory flotation tests.

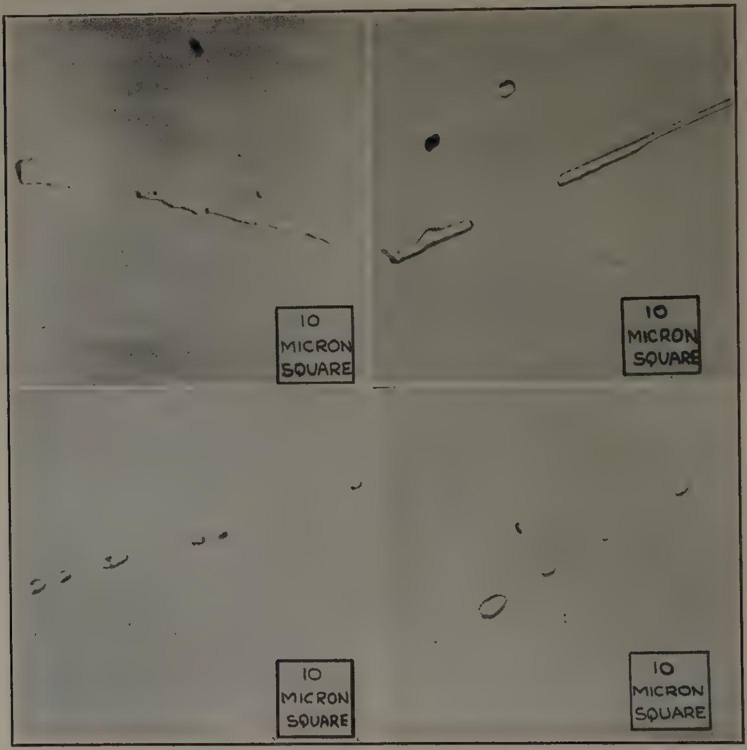

FIG. 1.—VEINLET OF GOLD IN PYRITE CRYSTAL. × 1000.
Width of vein and diameter of two small particles above it can be judged from size of 10-micron square.

FIG. 2.—ANOTHER EXAMPLE OF VEINLET OF GOLD IN PYRITE. × 1000.

FIG. 3.—SERIES OF GOLD PARTICLES COMPLETELY SURROUNDED BY PYRITE. × 1000.

FIG. 4.—ANOTHER EXAMPLE OF GOLD PARTICLES IN PYRITE. × 1000.
Diameter of smallest particle in group is about 2000 times that of a unit crystal of gold. Determined from data given by R. W. G. Wyckoff. [The Structure of Crystals, 236. New York, 1924. Chemical Catalog. Co.]

2. Dr. P. Price, now a member of the company's geological staff, carried out a great deal of work both at Noranda and McGill University to determine by microscopic means the nature of the gold occurrence in Noranda ore. This work is touched on in a paper by Dr. Price,[3] and

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[3] P. Price: Geology and Ore Deposits of the Horne Mine, Noranda, Quebec. *Canadian Min. and Met. Bull.* 263 (1934) 108.

the photomicrographs presented herewith (Figs. 1 to 6, 8 and 11 to 14) demonstrate the practical value of this work in connection with the metallurgical problem.

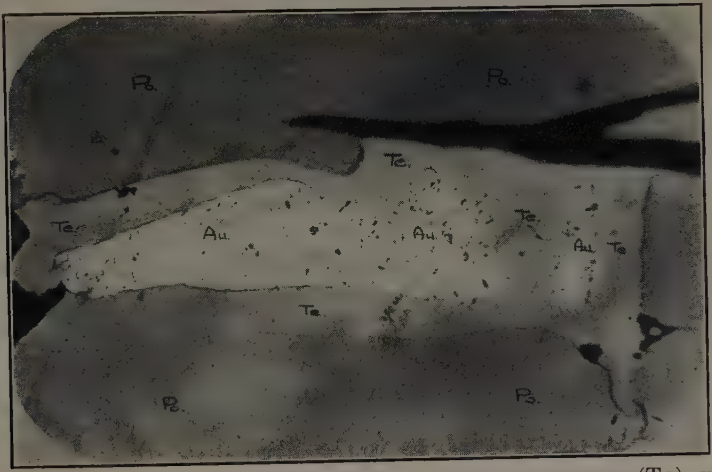

Fig. 5.—Gold (Au) completely surrounded by gold telluride (Te), which in turn is bounded by pyrrhotite (Po). × 950.

Typical of gold occurrences in pyrrhotite. So far, however, gold particles completely sheathed in telluride have not been encountered in pyrite. Note also relatively larger size of gold in pyrrhotite than in pyrite.

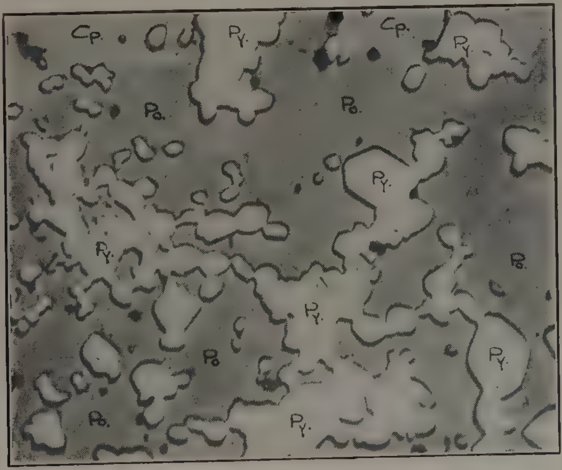

Fig. 6.—One type of concentrating ore characterized by extremely intimate replacement of pyrite (Py) by pyrrhotite (Po) and chalcopyrite (Cp). × 1000.

While ore represented by this photograph does not predominate in the mill feed, its tonnage is nevertheless appreciable.

3. An investigation was undertaken in the concentrator laboratory into the sizing of fine material, which resulted in the development of a method by means of which five size zones of constant range were estab-

lished below 325 mesh with the aid of a Nobel elutriator. (See Appendix 1.)

4. Members of the concentrator research staff investigated the quantitative determination of pyrite, pyrrhotite and magnetite by chemical means and developed a method by which the percentage of each of these minerals, when present with chalcopyrite in the ore and various mill products, could be determined rapidly with a degree of accuracy greatly exceeding that ordinarily obtained by making mineral counts with the aid of a microscope. (See Appendix 2.)

While these investigations were in progress the general review of the problem that had been undertaken confirmed the necessity of recovering the pyrite from the copper-circuit tailing and re-treating it separately as a necessary step toward improving gold recovery.

The work of floating and re-treating the pyrite, which already had been carried out, gave a very fair idea of the metallurgical results to be expected by adopting this procedure, and the problem that remained was primarily concerned with determining whether the value of the gold recovered would exceed the cost of its recovery.

In connection with this point it might be well to mention that when this investigation into increasing gold recovery was undertaken the prospect of gold advancing in value to a figure in excess of $30 an ounce was not entertained, consequently the economic problem was based on gold being worth $20 an ounce. In order that an idea may be gained of the economic limits thus imposed, the following summary may prove of interest. At the time the investigation was started the average copper-circuit tailing carried 0.055 oz. of gold per ton. This tailing contained 15.6 per cent pyrite, which assayed 0.17 oz. of gold per ton of pyrite. This meant that the gold content of the nonpyritic portion of the tailing was 0.033 oz. per ton. As the nonpyritic portion of the tailing consisted very largely of pyrrhotite and the magnetic sorting tests had shown that when the pyrrhotite was ground to minus 200 mesh it carried about 0.02 oz. of gold per ton, the difference between this figure and 0.033 oz. was taken as representing gold that could be recovered from the pyrrhotite if the necessary degree of fineness were obtained.

In the preceding paragraph the gold associated with the pyrite in the mill tailing was given as 0.17 oz. per ton of pyrite. In practice, however, a concentrate containing 100 per cent pyrite could not be produced and laboratory test work showed that it would not be advisable to anticipate a concentrate carrying more than 85 per cent pyrite if a high recovery of this mineral were to be made. This test work also showed that the nonpyritic portion of the pyrite concentrate consisted almost entirely of pyrrhotite, which meant that the average grade of pyrite concentrate produced would contain about 0.15 oz. of gold per ton.

The re-treatment tests made in the laboratory on the pyrite concentrate showed that a recovery of between 45 and 50 per cent of the gold associated with the pyrite could be expected by flotation following fine grinding. This recovery, with gold at $20, represented a gross return of about $1.40 per ton of pyrite. The cost of floating the pyrite from the mill tailing, together with the cost of its subsequent re-treatment by fine grinding and flotation had, therefore, to be kept within this figure and still leave a sufficient margin to amortize and yield a return on the capital required to provide the necessary equipment for the re-treatment step.

To complicate the picture still further, it was found that the pyrite concentrate floated from a certain type of ore assayed 0.11 oz. of gold per ton, which meant that the gold recovered from this material would have a value of only about $1.05 per ton. This figure, therefore, was selected as the limit within which the cost of producing and re-treating the pyrite concentrate had to be kept.

An idea of the nature of the work imposed by this limit can be gained from a consideration of the following points:

1. To make an accurate estimate of the cost of floating a ton of pyrite, it was necessary to know not only the cost of treating a ton of copper-circuit tailing, but also the probable tonnage of pyrite concentrate that would be produced. The cost of treating a ton of copper-circuit tailing was determined very closely by interpreting laboratory results in terms of known mill costs, but owing to the variableness of the pyrite content of the ore it was deemed necessary to run pyrite assays on monthly mill composite samples back to the beginning of 1931, in order to be reasonably sure of the average tonnage of pyrite that would be floated.

2. It was necessary to estimate as accurately as possible the cost of regrinding this pyrite concentrate. This estimate was made by applying a method developed by Gross and Zimmerley[4] by means of which the unit cost of surface formation in the main tertiary grinding circuit was determined. The figure so obtained was then used to evaluate the cost of surface formation called for in the laboratory grinding tests. This method, which we have found of definite value in determining the economic limits of grinding, is discussed in greater detail later in this paper.

3. The correct conditions of alkalinity and aeration had to be established for the flotation circuit used, in order to recover the gold liberated from the reground pyrite.

4. As the best recovery of gold, with a satisfactory ratio of concentration, made by flotation from reground pyrite was only 45 to 50 per cent, and further grinding to improve recovery did not promise to be profit-

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[4] J. Gross and S. R. Zimmerley: Efficiency of Grinding Mills. U.S. Bur. Mines *Rept. of Investigation* 2952 (1929).

able, some means, if possible, had to be found to improve results, and with this object in view tests were undertaken to determine whether some of the gold that still remained with the re-treated pyrite residue could be extracted profitably from it by cyanidation. This investigation into the possibility of cyaniding the reground pyrite residue presented four points that required special consideration, three of which were directly connected with the fineness to which it had been ground preparatory to the preceding flotation step. The four points were:

a. The material to be cyanided carried between 0.08 and 0.10 oz. of gold per ton.

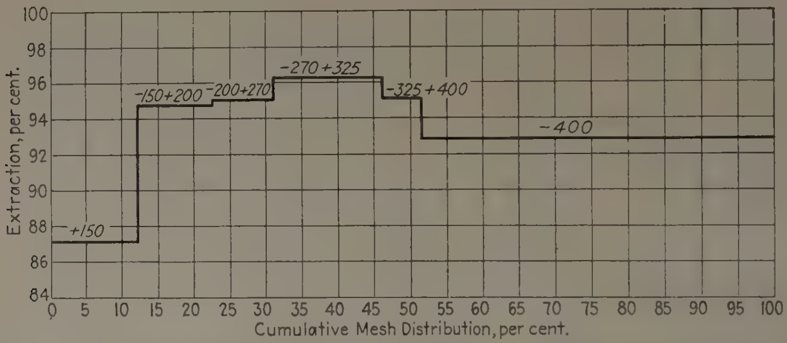

FIG. 7.—DISTRIBUTION AND RECOVERY OF COPPER AS CHALCOPYRITE IN VARIOUS MESH ZONES.

Area above recovery line represents proportion of copper lost, while if scale were retained and graph completed below the line the resulting area would represent the proportion of copper recovered.

b. This material contained about 15 per cent pyrrhotite, which in this case was a more virulent cyanicide than usual because of its extreme fineness.

c. Some chalcopyrite still remained with the pyrite after it had been reground and refloated and this chalcopyrite, although only a very small percentage was present, was a very serious cyanicide because of the large surface presented.

d. Because of the extremely fine grinding, dewatering and washing the final residue presented several difficulties.

The points listed give an idea of the nature of the problem involved in cyaniding this product, and though pilot results recently have justified the approval of an appropriation for building a unit (which will be placed in operation early in 1935), to cyanide the entire pyrite residue, it is felt at the present time that definite statements with regard to this treatment step would be premature.

When the laboratory work connected with segregation of the pyrite and its re-treatment began to take definite shape, a contemporaneous investigation was undertaken to determine whether additional gold could be recovered from the pyrrhotite. As the magnetic portion of the mill tailing which was finer than 200 mesh carried about 0.02 oz. of gold per ton and was almost entirely pyrrhotite, it seemed reasonable to assume that this represented the figure to which the gold content of the pyrrhotite could be reduced by flotation.

Cyanide tests on the pyrrhotite also showed a high recovery of gold but a consumption of lime and cyanide that was prohibitive. Flotation tests, however, yielded almost immediate results with a high ratio of concentration as soon as the necessary fineness of grinding was employed.

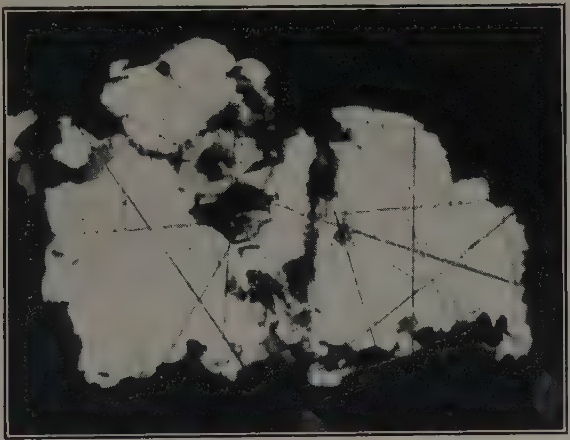

Fig. 8.—Particle of gold recovered by gold blankets. × 140.

This particle has been mounted in bakelite and the way in which it is built up out of several smaller particles can be clearly seen. Note also the relatively low magnification.

In view of these last results, the alternative was presented of carrying the copper-circuit grind to a point where practically all the material in that circuit had been reduced to minus 200 mesh, or of maintaining a relatively coarse grind in the copper circuit and grinding the pyrrhotite to its required ultimate fineness after the chalcopyrite and pyrite had been removed.

A graph reproduced in Fig. 7, showing the recovery of copper in the various zones of sizing, will explain the reason for selecting the second of these alternatives, as it will be seen that the recovery of chalcopyrite begins to fall off when it has been ground finer than 325 mesh.

Recovery of Unfloated Free Gold

Intimation has already been given, and will later be supported by figures, showing that a high recovery of free gold is made by flotation. Notwithstanding this fact it was found that there were some particles of free gold in the copper and pyrite circuit tailings, which were made up of smaller particles that had been hammered into aggregates in the grinding mills and had become too large to float.[5] In order that these large particles might be recovered, it was decided to install blankets in the launder that carried the pyrite-circuit tailing to the pyrrhotite section. (See Fig. 8.)

Discussion of Mill Results

The development of the pyrite and pyrrhotite re-treatment steps in practice, and the relation they bear to the main copper circuit, can be seen in the mill flow sheet (Fig. 9), while in Table 1 the metallurgical results obtained in the entire mill and in the various individual circuits for the months of April to July, inclusive, for 1934 are given. These show that during the period under review 92.77 per cent of the copper in the mill heads was recovered in the copper-circuit concentrate and this recovery was increased to 96.90 per cent by the copper floated in the pyrite and pyrrhotite re-treatment steps. As a partial counter, however, to this increase in recovery, the additional tonnage of concentrates produced by these two circuits, when combined with the copper-circuit concentrate, reduced the over-all mill ratio of concentration to 5.24 into 1. This last ratio, however, was higher than that at which the mill had been operated before the introduction of the two re-treatment steps, so it will be seen that the predicted requirement regarding the ratio of concentration had been met satisfactorily.

Table 1.—*Metal Recoveries and Ratios of Concentration*
(Based on Mill Heads)

Circuit	Recovery, Per Cent		Ratio of Concentrate
	Copper	Gold	
Main copper circuit	92.77	65.68	6.34
Pyrite regrind circuit	3.08	6.12	66.6
Pyrrhotite regrind circuit	1.05	7.33	56.3
Over-all	96.90	79.13	5.24

Table 1 also shows that the recovery of gold made in the copper circuit was only 65.68 per cent, and that this figure was augmented by 13.45 per cent as a result of additional gold subsequently recovered from the pyrite and pyrrhotite.

This recovery, however, still left 20.87 per cent of the gold in the mill tailing, and as such a loss, though less than formerly made, is nevertheless a serious one, an analysis showing the reason for its existence may be in order.

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[5] E. S. Leaver and J. A. Woolf: Flotation of Metallic Gold—Relation of Particle Size to Floatability. U.S. Bur. Mines *Rept. of Investigation* 3226 (1934); also *Eng. & Min. Jnl.* (1934) **135**.

Pyrite and Pyrrhotite Flotation Circuits

The mill flow sheet (Fig. 9) shows that the tailing from the copper circuit enters the pyrite flotation circuit, where the pyrite is recovered for re-treatment. In Table 2 the metallurgical performance of this circuit for the same period as that covered by Table 1 is given. These results show that 43.5 per cent of the total gold lost in the copper-circuit tailing was recovered in the pyrite concentrate, and as will be seen from Table 3, this recovery was further augmented by re-treating the pyrite circuit tailing in the pyrrhotite circuit. The increase in gold recovery effected in the pyrrhotite re-treatment step was equivalent to an additional recovery of 21.8 per cent of the gold in the copper-circuit tailing. This additional recovery, when added to the gold recovered in the pyrite concentrate, gave a total recovery of gold made from the copper-circuit tailing of 65.3 per cent, a figure which in terms of mill heads represented a total additional recovery of 22.4 per cent. The difference between this last figure and the figure of 13.45 per cent, which was reported as the percentage of gold recovered in the pyrite and pyrrhotite re-treatment steps, represents the gold lost when the pyrite concentrate is reground and only treated by flotation.

Table 2.—*Metallurgical Results, Pyrite Flotation Circuit*

	Assays			Percentage of Recovery in Terms of Copper Circuit Tailing			Ratio of Concentrate
	Pyrite, Per Cent	Copper, Per Cent	Gold, Oz.	Pyrite	Copper	Gold	
Heads	15.2	0.20	0.04	78.8	50.4	43.5	7.1
Concentrate	83.8	0.72	0.148				
Tailing	3.8	0.12	0.032				

Table 3.—*Metallurgical Results, Pyrrhotite Re-treatment Circuit*

	Assays		Percentage of Recovery in Terms of				Ratio of Concentrate in Terms of	
			Pyrrhotite Circuit		Copper Circuit Tailing			
	Cu, Per Cent	Au, Oz.	Cu	Au	Cu	Au	Pyrrhotite Circuit	Copper Circuit Tailing
Heads	0.117	0.032	28.4	38.4	14.1	21.8	40.3	47.1
Concentrate	1.34	0.495						
Tailing	0.088	0.020						

Table 1 shows that the copper-circuit tailing before re-treatment contained 34.32 per cent of the gold in the ore. It has been shown that the gold that entered the pyrite and pyrrhotite concentrates was equivalent to 22.4 per cent of the gold in the ore. Consequently, as these last two circuits are operated in series, the difference between the foregoing figures of 11.92 per cent represents the gold that remained in the pyrrhotite-circuit residue. An analysis of this loss follows.

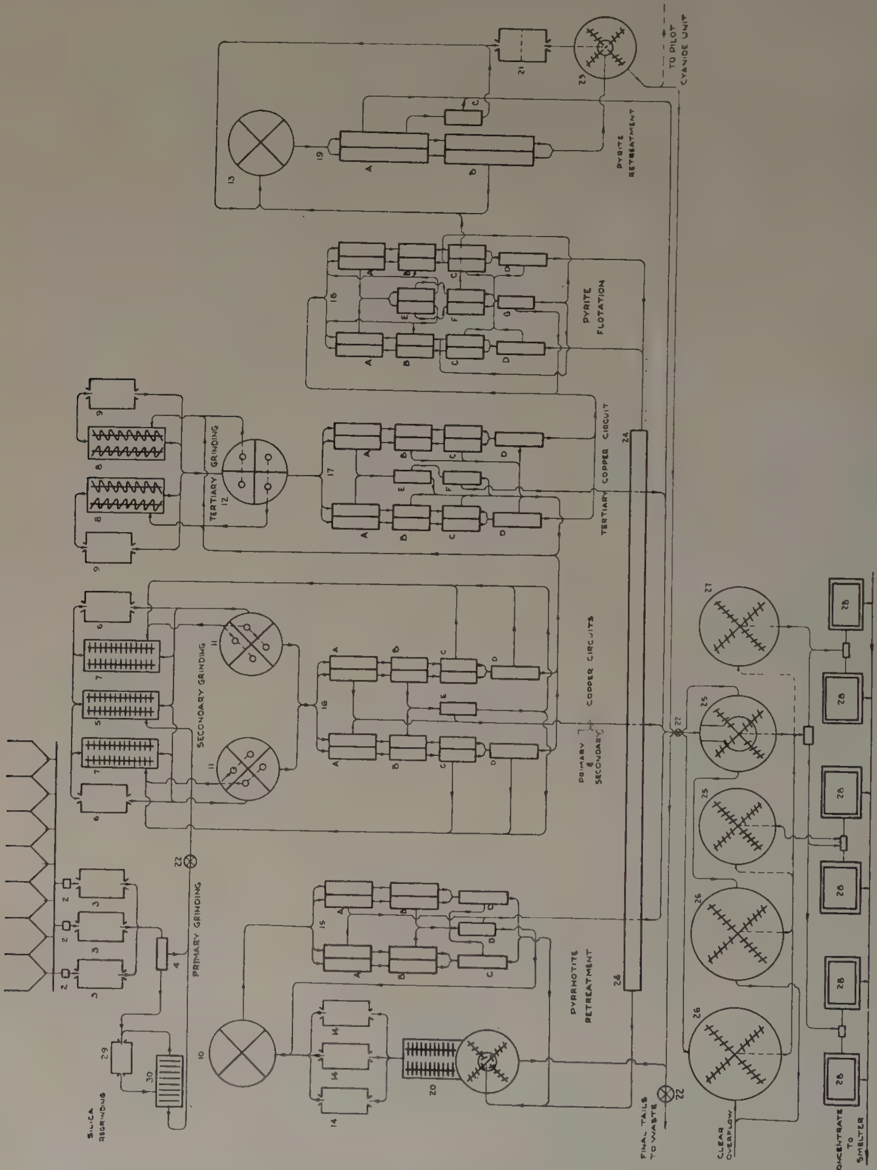

FIG. 9.—For caption see opposite page.

From Table 2 it will be seen that the pyrrhotite-circuit head, which is the pyrite-circuit tailing, carried 3.8 per cent pyrite, which was not recovered in the pyrite-circuit concentrate. The gold associated with this pyrite can be calculated therefore from Table 2, if it is assumed that the nonpyritic portion of the pyrite concentrate shown in that table is pyrrhotite having a gold content of 0.025 oz. per ton. The reason that the gold value of this pyrrhotite has been taken as 0.025 oz. per ton instead of 0.02 oz. per ton is that, as the pyrite circuit precedes the pyrrhotite circuit, the pyrrhotite that enters the pyrite concentrate has not been ground to its ultimate fineness.

From the data above, it will be found that a unit of pyrite carries 0.00172 oz. of gold, and consequently the 3.8 per cent pyrite that entered the pyrrhotite circuit carried with it 0.0065 oz. of gold per ton of entering feed. Some of this gold would be recovered, however, from the pyrite following its regrinding in the pyrrhotite circuit, but as the pyrrhotite grinding step is not carried as far as the pyrite regrinding step the recovery of the gold from the tramp pyrite would be only about two-thirds of that made by regrinding the pyrite concentrate proper. The last-named operation is still to be discussed, but Table 4 will show that the gold recovery effected from the pyrite concentrate after it had been reground was 41.56 per cent, consequently a figure of 27 per cent has been taken as representing the gold recovered from the pyrite that entered the pyrrhotite circuit. Such being the case, of the 0.0065 oz. of gold per ton of feed introduced into the pyrrhotite circuit by the tramp pyrite, 0.0047 oz. escaped into the final tailing. Taking into account the ratio of concentration made in the pyrrhotite circuit, this caused a gold loss of 0.0048 oz. per ton of pyrrhotite tailing.

In determining the gold lost with the pyrrhotite, an analysis of the composite mill tailing for the period under consideration showed that it contained 63.5 per cent pyrrhotite, and a magnetic sorting test made on the same material gave a gold value of this pyrrhotite of 0.021 oz. per ton. The gold, therefore, associated with the pyrrhotite in a ton of pyrrhotite-circuit tailing was 0.013 oz.

FIG. 9.—FLOW SHEET OF CONCENTRATOR, NORANDA MINES, LIMITED.

1. Ore bins
2. Weightometers
3. Primary ball mills
4. Trommels
5. Primary classifier
6. Secondary ball mills
7. Secondary classifiers
8. Tertiary classifiers
9. Tertiary ball mills
10. Pyrrhotite re-treatment aerator
11. Primary } copper circuit
    Secondary } aerator
12. Tertiary copper circuit aerator
13. Pyrite re-treatment aerator
14. Pyrrhotite regrinding ball mills
15. Pyrrhotite re-treatment flotation
    A. First rougher cells
    B. Second rougher cells
    C. Tailing cells
    D. Cleaner cell
16. Primary and secondary copper flotation
    A. First rougher cells
    B. Second rougher cells
    C. Third rougher cells
    D. Tailing cells
    E. Cleaner cell
17. Tertiary copper circuit
    A. First rougher cells
    B. Second rougher cells
    C. Third rougher cells
    D. Tailing cells
    E. First cleaner cell
    F. Final cleaner cell
18. Pyrite flotation circuit
    A. First rougher cells
    B. Second rougher cells
    C. Third rougher cells
    D. Tailing cells
    E. First cleaner cell
    F. Final cleaner cell
    G. Third cleaner cell
19. Pyrite re-treatment flotation
    A. First rougher cell
    B. Second rougher cell
    C. Cleaner cell
20. Quadruplex bowl classifier
21. Pyrite regrinding ball mill
22. Automatic samplers
23. Noranda classifier
24. Blanket launder
25. Thickening and conditioning tank
26. Double deck thickening tank
27. Thickened pulp storage tank
28. Filters
29. Ball mill
30. Drag classifier

The combined loss of gold in the pyrrhotite-circuit tailing due to entrainment with the pyrite and pyrrhotite was therefore 0.0178 oz. per ton. Table 3, however, shows that the total gold lost in the pyrrhotite-circuit tailing was 0.02 oz. per ton. The difference, therefore, between these two figures, which is 0.0022 oz., may be taken as representing the loss due to free gold, which failed to float. Therefore, as the gold lost in the pyrrhotite tailing represents 11.92 per cent of the gold present in the ore, this percentage can be divided as follows:

	Per Cent
Gold lost with the pyrrhotite	7.75
Gold lost with unrecovered pyrite	2.86
Loss of free gold, which failed to float	1.31

Individually none of these gold losses would be serious but collectively they make a formidable total to which must also be added the gold lost in the re-treatment of the pyrite-circuit concentrate.

Pyrite Regrinding Circuit

It has been stated in the preceding pages that the gold content of the pyrite concentrate is relatively low and fine grinding is necessary in order to liberate the gold which it carries. Table 4 shows the results obtained by re-treating the pyrite concentrate produced from the copper-circuit tailing during the four-month period under review. The circuit employed for obtaining these results is depicted diagrammatically in the general mill flow sheet (Fig. 9). The sequence of treatment was, and at the time of writing is, as follows: The pyrite concentrate floated from the copper-circuit tailing is laundered to an aerator where it is aerated for 40 min. with lime. From the aerator it overflows to flotation cells, and the material that floats is pumped back to join the main copper-circuit concentrate. The tailing from these flotation cells then goes to a classifier of local design and the overflow from this classifier is the pyrite re-treatment circuit tailing. The underflow of the classifier is removed from it by diaphragm pumps, and constitutes the feed to the pyrite-regrinding mill.

Table 4.—*Metallurgical Results, Pyrite Regrinding Circuit*

	Assay		Recovery Based on				Ratio of Concentrate Based on	
			FeS_2 Circuit		Cu Tailing			
	Cu, Per Cent	Au, Oz.	Cu	Au	Cu	Au	FeS_2 Circuit	Cu Tailing
Heads	0.72	0.148	81.98	41.56	41.32	18.10	7.9	56.6
Concentrate	4.66	0.490						
Tailing	0.14	0.096						

After being reground it is pumped to the aerator, which it originally entered, and again, after a second 40-min. aeration period, overflows to flotation, and the portion that does not float is returned to the classifier for reclassification. Here the fine material is again eliminated while the coarse remains in the circuit until it either enters the flotation concentrate or is ground fine enough to overflow the classifier.

It might appear that the adoption of the foregoing flow sheet would necessitate a large increase in the number of flotation cells required to take care of the circulating load of the grinding mill. This circulating load in terms of the entering feed varies between 300 and 400 per cent, and the pilot unit tests showed that ample capacity for its treatment could be provided by merely doubling the effective depth of the flotation cells used. The reason that this was the only change made necessary was that in the closed-circuit operation the density of the pulp passing through the flotation cells was twice that of the classifier overflow, and consequently accounted for half the increase in capacity needed to provide a flotation period equivalent to that of the open circuit. The effect of closed-circuit flotation on the coarseness of the concentrate produced can be seen from the figures given in Table 5. The reasons for adopting this flow sheet were as follows:

TABLE 5.—*Size Distribution, Tail and Concentrate of Pyrite Regrinding Circuit*

Mesh	+100	−100 + 200	−200 + 325	−325	Total
Concentrate	1.7	4.5	10.9	82.9	100.0
Tailing		0.4	4.3	95.3	100.0

1. A better recovery of copper could be made by giving the chalcopyrite a chance to float before, instead of only after, the regrinding operation. (For the effect of fine grinding on copper flotation see Fig. 7.)
2. The aeration period required for conditioning the entering feed was found to be the same as that necessary to recondition the ball-mill discharge.
3. Placing the flotation cells in closed circuit with the mill obviated the necessity of duplicating the aerator and flotation equipment required to treat the incoming concentrate prior to grinding.
4. Closed-circuit flotation gave the gold a chance to float as soon as it was ground free and reduced the possibility of overgrinding it.

The effect of closed-circuit grinding and the correct degree of aeration on the gold recovery made by flotation is shown by Table 6, which gives the results obtained during two different periods in 1933 when a pyrite re-treatment unit for 200 tons per day was being operated on different flow sheets for pilot purposes. The flotation, classifying and regrinding equipment used in each of the periods was identical. In the first period, however, an open circuit with 20 min. aeration prior to flotation was used, while in the second the circulating load of the mill was floated after being aerated in an aerator which had sufficient capacity to give the pulp 40 min. aeration before flotation.

TABLE 6.—*Results during Two 24-hour Periods*

	Copper, Per Cent			Gold, Oz.			Extraction, Per Cent		Ratio of Concentrate
	Heads	Conct.	Tails	Heads	Conct.	Tails	Cu	Au	
Open-circuit flotation with 20 min. aeration, July 1–12, 1933	0.25	1.13	0.14	0.16	0.29	0.14	49.3	22.7	10.3
Closed-circuit flotation with 40 min. aeration, July 20–31, 1933	0.30	1.68	0.09	0.16	0.51	0.11	70.3	42.6	8.0

From an experimental standpoint it is unfortunate that the change to closed-circuit flotation and the increase in the aeration period were made contemporaneously, but the mill extension program was being held in abeyance till satisfactory pilot-mill results were obtained, and as laboratory tests had shown that both changes were desirable the effect produced by each was not investigated. It is interesting to record, however, that in spite of the increased tonnage that went through the flotation circuit, due to treatment of the mill circulating load, the amount of collecting agent used per ton of new feed was less than 33 per cent of that employed during the first period. This result we attribute to lengthening the time of the aeration step, as a similar reduction in reagent consumption took place following the introduction of aeration into the main copper circuit in 1929.

It has been found also, in connection with pyrite re-treatment, that when the correct period of aeration has been determined the lime content of the grinding mill should be carried at or near saturation. If this is done the recovery of gold and also the grade of concentrate subsequently made by flotation is materially improved.

The foregoing points have been found to be of primary importance in recovering gold by flotation from Noranda's reground pyrite and for convenience they may be summarized as follows:

1. After the pyrite has been reground the gold will not float properly until the pulp has been aerated to the correct degree.
2. When the correct aeration period has been established the use of a high-lime grinding circuit rather than a low-lime circuit improves subsequent flotation recovery, and ratio of concentration.

3. After liberation, the gold should be given a chance to float as soon as the correct condition for its flotation has been established.

The only other point of especial interest in connection with this circuit is that of classification. Because of the fineness to which the pyrite must be ground, it was decided to develop a classifier combining the principles of a thickener and a cone, and to equip it with bottom-driven rakes. The coarse material in the pulp fed to this classifier is removed from the bottom part of the cone by diaphragm pumps at a density of about 80 per cent solids and goes directly to the regrinding mill, while the finished material constitutes the tank overflow. The operation of this machine is extremely simple. The sizing figures given in Table 7 give typical results obtained with this classifier, while handling its normal circulating load of about 350 per cent.

TABLE 7.—*Classifier Results*

	Distribution, Per Cent						
Mesh	+200	−200 +325					
Average size, microns	74	58	36	30	23	15	8
Pyrite concentrate	35.3	20.8	22.6	7.3	5.5	4.2	4.3
Classifier feed	20.4	23.8	32.7	6.7	1.7	4.0	10.7
Classified underflow	27.6	35.6	29.1	1.0	0.7	1.3	4.7
Classified overflow		4.7	42.2	18.2	3.8	8.5	22.6

TABLE 8.—*Average Gold Content of Pyrite*

	Gold per Ton, Oz.							
Mesh	+100	−100+150	−150+200	−200+325				
Average size, microns	147	125	89	58	36	30—23[a]	15	8
Pyrite concentrate								
Before flotation re-treatment	0.20	0.18	0.15	0.14	0.13	0.12	[b]	[b]
After flotation re-treatment				0.14	0.13	0.12	0.11	0.14
After cyaniding				0.06	0.05	0.04	0.03	0.02 0.025[c]

[a] For the purpose of assay the 30 and 23-micron products were combined.

[b] Owing to the small proportion of material present, these products were not assayed.

[c] On some ore a 0.02-oz. residue is made consistently, whereas on other ores it is difficult to bring it below 0.025 oz.

The importance of fine grinding and proper classification in the treatment of the pyrite can be seen from Table 8, which shows the average gold content of the pyrite in different size zones during its re-treatment.

Pyrite Regrinding Circuit, Surface Formation

An idea of the surface formed in grinding the pyrite concentrate to the fineness of the classifier overflow shown in Table 7 will be gained from Table 9, which has been developed from the sizing data given in Table 7 by applying a formula[6] developed by Gross and Zimmerley. From the figures given in Table 9, it will be seen that the amount of new surface formed in regrinding a ton of pyrite concentrate was 520.4 × 10^6 sq. cm. per ton. This area actually exceeds that formed in all the previous stages

TABLE 9.—*Surface Formation, Pyrite Regrinding Step*

Average Size of Particles, Microns	Surface, Sq. Cm. per Ton × 10^6	Pyrite Concentrate Before Regrinding		Pyrite Concentrate After Regrinding	
		Per Cent Weight	Product Per Cent Weight × Surface = Sq. Cm. × 10^6 per Size Zone	Per Cent Weight	Per Cent Weight × Surface = Sq. Cm. × 10^6 per Size Zone
74	151	35.3	53.3		
58	356	20.8	74.0	4.7	16.7
36	568	22.6	128.4	42.2	239.7
30	665	7.3	48.5	18.2	121.0
23	835	5.5	45.9	3.8	31.7
15	1,232	4.2	51.7	8.5	104.7
8	2,232	4.3	96.0	22.6	504.4
Total surface			497.8		1,018.2

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[6] The formula used was that given in reference of footnote 4, where (p. 3) it is stated that the measured surface for quartz in square centimeters per gram of quartz cubes, or spheres, is equal to $\frac{22{,}600}{d} \times R$. In this formula $\frac{22{,}600}{d}$ equals the theoretical surface per gram of quartz in square centimeters when d represents the average diameter of the measured particles in microns, while R is the ratio of measured to theoretical surface. The value for R for a 38-micron particle is given as 1.98 and for a 1.5 micron particle as 1.64, while the factor used to convert areas for quartz to those for particles of pyrite of similar size is given as 0.96. As, however, pyrite is heavier than quartz, the surface area of a gram of pyrite particles is less than that of a gram of quartz particles of the same size. This variation can be taken care of by multiplying the expression for surface area by the specific gravity of quartz and dividing by that for pyrite. If this is done the surface area of pyrite in square centimeters per ton of pyrite may be determined by making the necessary substitutions in the following formula:

$$\frac{2000 \times 454 \times 0.96 \times 22{,}600 \times R \times \text{sp. gr. } (SiO_2)}{d \times \text{sp gr. } (FeS_2)}$$

For additional related papers by Gross and Zimmerley see *Trans.* A.I.M.E. (1930) **87**, 7, 27, 35.

of comminution through which the pyrite had passed before entering the pyrite regrinding circuit.

At first glance this calculation for the new surface formed may seem purely academic. It might be of interest, however, to repeat the statement made earlier in this paper, that in the laboratory work carried out before the 200-ton pilot unit was placed in operation in 1933 a similar set of calculations was prepared to determine the surface produced in the laboratory tests, and the figure so obtained was compared with the surface produced in the tertiary grinding mill of the copper circuit. As the grinding cost for this last-named operation was known, a figure for the cost of regrinding the pyrite was obtained which checked the subsequent actual cost for regrinding pyrite to within two cents per ton.

A further example of the application of this method for determining grinding costs and economic grinding limits may be of interest. Let it be supposed that the question is asked whether, if the whole pyrite residue were being cyanided, it would pay to grind the 58-micron and 36-micron material down to an average size of 8 microns. The method of making the calculation would be as follows: In Table 8, the gold value of 58-micron material after cyaniding is given as 0.06 oz. per ton, and that for 36-micron material as 0.05 oz. per ton. The percentages of these sizes in the classifier overflow, as given in Table 7, are 4.7 and 42.2 per cent, respectively. Table 8 also shows that the gold content of 8-micron pyrite is 0.02 or 0.025 oz. per ton. In making an estimate such as that called for in the present case it would be advisable to take the gold content of the 8-micron residue as the higher of these two figures, while the gold value of the coarse material before further treatment, on account of the small proportion of 58-micron material present, would be taken as 0.05 oz. per ton. Such being the case, the additional recovery of gold to be expected from the increased fineness of grinding would be 0.025 oz. per ton of original coarse material. With gold at $30 per ounce this would represent an added saving of 75¢ per ton of material reground. As, however, there would only be 0.469 tons of this material per ton of total residue, the over-all saving would be only 35¢. The original surface of the 0.469 tons of coarse material will be found, from Table 9, to be 256×10^6 sq. cm., while its surface if ground to 8 microns would be $2232 \times 10^6 \times 0.469 = 1047 \times 10^6$ sq. cm. This increase in surface would represent a formation of new surface area of 791×10^6 sq. cm.

The surface formed in regrinding a ton of pyrite concentrate has been shown to be 520.4×10^6 sq. cm., and if the cost of this operation is taken as 24¢ per ton, it will be found that the probable cost of producing an area equal to 791×10^6 sq. cm. will be 36¢. This figure is actually greater than the value of the gold that it might be expected to recover. Consequently, even allowing for considerable modification of the foregoing figures to provide for possible experimental errors, it would seem advisable to accept them as a warning that care would have to be taken not to exceed the economic limit of treatment in trying to obtain the desired increase in recovery.

Conclusion

In conclusion, it should be pointed out that owing to the length of time covered by this investigation it has not been possible to deal with all its phases in equal detail, and as a result certain portions of the work have been dealt with more fully than others.

In regard to the cyanide step, the possibilities of which have only been touched on, the omission of detail has been intentional, because we are still learning so much about this phase of the operation that information regarding it would be premature.

Acknowledgments

The investigation described on the foregoing pages shows clearly that in spite of the progress made in improving the gold recovery from Noranda's massive sulfide concentrating ore a great deal of work still remains to be done. For this reason it is a particular pleasure to thank the management and board of directors for permitting the publication of this paper at the present time, and also for the help given and sympathetic consideration shown during the period that the investigation herein described has been in progress.

It is also a pleasure to acknowledge the assistance that has been given us by the Canadian Bureau of Mines, under its chief, Mr. W. B. Timm, and Dr. M. W. Haycock of his department is to be congratulated on the method he has developed for embedding and polishing the various finely ground products which we have sent him. These products have been returned to us ready for microscopic examination and this work, together with that of preparing and examining the ore sections illustrated, has been done by Dr. P. Price, of the Noranda geological staff, to whom reference has been made in the preceding pages, and without whose assistance the photomicrographs reproduced could not have been obtained.

As regards the work carried out in the concentrator laboratory, special mention should be made of the teamwork and determination shown by the mill research staff throughout the entire investigation in the face of frequently discouraging results. Much of the credit for setting this high standard of teamwork goes to F. J. Martin, now assistant mill superintendent at the Beattie Gold Mines and for several years a member of the mill staff at Noranda. With regard to the working out of the pyrite and pyrrhotite re-treatment steps, primary credit goes to T. R. Wearing and H. L. Ames, who first joined the company's staff in 1930 and who have since been actively engaged in the development of these steps both in the laboratory and in the mill. It is a pleasure to acknowledge

also the work done by B. Robinson in initiating the method of pyrite analysis and for carrying out many of the original experiments connected with elutriation and optical and magnetic sorting. This work has been carried on and developed by R. J. Morton, to whom credit is also due for first demonstrating the application of surface measurement figures to our grinding problem.

APPENDIX 1.—Sizing Finely Ground Pyrite with the Aid of a Nobel Elutriator*

The Nobel elutriator, a diagrammatic sketch of which is shown in Fig. 10, consists of a set of closed conical jars of increasing diameter operated in series under a constant pressure head of water, and connected by curved tubes in such a way that the feed is introduced into the bottom of the first jar, the overflow of which becomes the inflow at the bottom of the second, and so on.

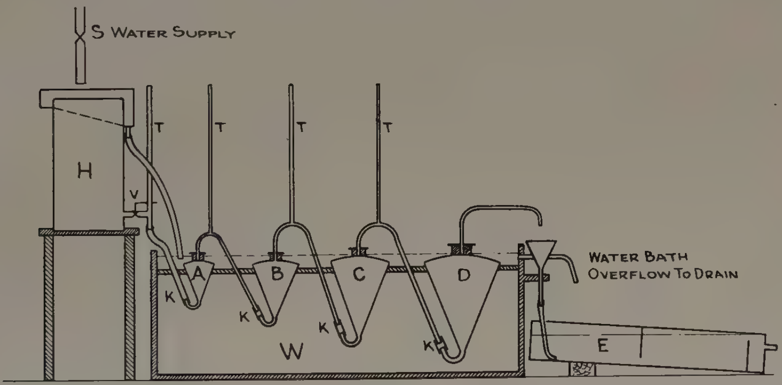

Fig. 10.—Diagrammatic section of Nobel elutriation arrangement as used at Noranda.

A. First elutriation vessel
B. Second elutriation vessel
C. Third elutriation vessel
D. Fourth elutriation vessel
E. Large pan for fifth elutriation product
H. Constant head tank
K. Rubber connections
S. Water supply to constant head tank
T. Copper pipe connections with air vents
V. Valve control
W. Water bath

At first the results obtained with this apparatus were not satisfactory. The first difficulty was caused by air bubbles collecting in the bends of the glass tubes connecting the various jars. These bubbles prevented a steady flow of water and also had a decided tendency to carry oversize sulfide particles into the next jar and salt its product. This difficulty was overcome by replacing the glass tubes connecting the various jars with copper tubes which had long tubular air vents rising vertically from the apex of the bends where the air was collecting. With this improved connection the air bubbles were removed and a steady flow of water through the elutriator was obtained.

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* In presenting this appendix, the author wishes to acknowledge, in particular, the work of R. J. Morton, of the concentrator metallurgical staff, who not only played a major part in the development of the procedures here described, but also rendered material assistance in preparing them for publication.

The next point investigated was the effect of temperature, for the reason that the coefficient of viscosity of water between 15° and 25° C. varies over 2 per cent for each degree of temperature change. The importance of such a change can be judged from Stokes' law of viscous resistance, by means of which the terminal velocity of a small sphere falling freely under the influence of gravity in a viscous fluid can be expressed as follows:[7]

$$V = \frac{2}{9}a^2g \qquad \frac{(d_1 - d_2)}{\mu}$$

In this formula V equals the terminal velocity of the particle, a is its diameter, g the gravitational pull, d_1 and d_2 the specific gravity respectively of the solid particle and the liquid, and μ the viscosity of the liquid. It can be seen, therefore, that if a constant flow of water is maintained through the system each jar at its point of maximum cross-sectional area will have a different but constant value for V, and the various particles being sized will either remain or pass to the next jar, according to their diameter. If, however, comparable results are to be obtained for a series of runs μ must also have a constant value.

Such a condition would have necessitated the provision of equipment to provide constant thermostatic control, so that the water used could be brought to a uniform temperature for each run. Fortunately, however, an investigation into temperatures of tap water showed that, for our conditions, the increment of change in the temperature of the water during each run could be neglected, and it would only be necessary to vary V to take care of seasonal changes of μ in order to obtain comparable results between various series of tests, provided that changes in room temperature could be taken care of.

Changes in room temperature, accordingly, were provided for by almost completely immersing the bodies of the various jars in water in a metal trough mounted in a wooden casing, which acted as an insulating medium.

The complete set-up for the apparatus is given in Fig. 10, from which it can be seen that the water overflowing the small tank H furnishes the jar-immersion tank with a continuous supply of fresh water. Another advantage resulting from immersing the jars was the sealing of the copper outlet tubes at the points where they connect to the glass inlets of the respective jars. This advantage was unlooked for, but it was found that sealing the connections in this way greatly reduced the amount of entrained air that had to be eliminated through the air vents.

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[7] A. F. Taggart: Handbook of Ore Dressing, 551. New York and London, 1927. John Wiley & Sons and Chapman & Hall.

With the set-up as described, the procedure for elutriating a finely ground sample of pyrite is as follows: A sample weighing about 150 grams is placed in a small bottle containing 300 c.c. of water and ½ gram of powdered sodium silicate. The bottle and its contents are agitated by some suitable means for ½ hr., at the end of which time all the particle aggregates have been broken up and the sample fully dispersed.

The contents of the bottle are then transferred to vessel *A*, the water inlet of which is connected by a rubber tube to the outlet of the constant-head tank *H*. The flow of water from this tank is controlled by a valve *V*. The four elutriation vessels, *A*, *B*, *C* and *D*, which comprise the set are connected together as indicated in the diagram, and a long pan *E* is put in place, which takes the overflow from the last vessel *D*. This pan is sufficiently long to give a clear overflow if a suitable flocculating agent such as lime or zinc sulfate is added to it.

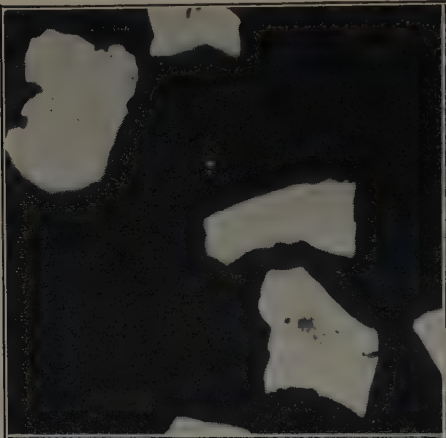
The elutriator is now started by opening the valve on the constant-head tank. Great care must be taken in doing this to avoid surges and our present practice is to open the valve gradually until a flow rate of about 150 c.c. per minute is obtained. This rate is maintained for a period of 2 hr., at the end of which time it is increased to 250 c.c. per minute, provided the test is being run at a standard temperature. Elutriation is then continued further at this rate for 22 hr. The rate of 250 c.c. per minute was decided upon as the standard for a temperature of 18° C. for the elutriation of reground pyrite concentrate. This material has a specific gravity of 4.8, and the rate of 250 c.c. was determined by experiment as that best suited to give a fair distribution in the different jars.

At the end of the elutriation period the content of each vessel is filtered and the residue dried and weighed. The distribution of the various-sized products is then calculated in per cent.

The determination of particle size in each container is ordinarily made only periodically, as we have found that if care is taken to insure that established conditions are maintained during the sizing period the average diameter of the particles found in each particular jar is a constant figure.

The method used for particle sizing is either by making a count of the particles dispersed on a slide, or by direct measurement of reflected particles which have been embedded and polished. This second alternative is particularly suitable for obtaining the size of particles in the finer products, as can be judged by referring to the photomicrographs (Figs. 11 to 14).

The only difficulty we still encounter with this apparatus is in obtaining corresponding jars that have the same cross-sectional area at the top. For some reason, jars are listed on a volumetric basis, instead of on a basis of maximum diameter, and as a result if a jar is broken and has to be replaced difficulty is frequently encountered in obtaining another to duplicate it. In spite of this difficulty, however, we believe it is better to use the glass jars than to substitute metal containers, which, though they would be unbreakable, would prevent inspection of the jars



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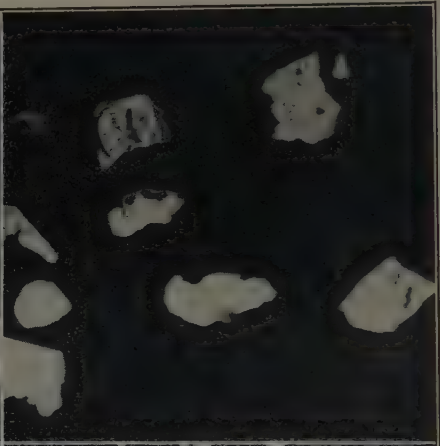

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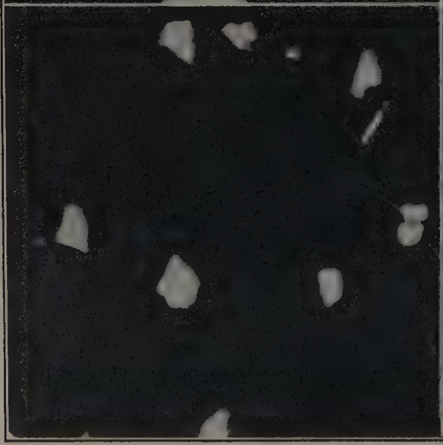

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FIG. 11.—PYRITE PARTICLES IN COARSEST ELUTRIATION PRODUCT. × 500.
These particles are mounted in bakelite, and their average size is about 36 microns; i.e. finer than 400 mesh.

FIG. 12.—PYRITE PARTICLES OBTAINED FROM COMBINING SECOND AND THIRD ELUTRIATION PRODUCTS. × 500.
Average sizes of these two products are, respectively, 30 and 23 microns.

FIG. 13.—PYRITE PARTICLES IN FOURTH ELUTRIATION PRODUCT. × 500.
Average size of this product is 15 microns.

FIG. 14.—FIFTH ELUTRIATION PRODUCT. PARTICLE SIZE ABOUT 8 MICRONS. × 500.
Absence of slime material in this product is characteristic of finely ground pyrite.

during and at the end of a run; a feature that we consider quite important in insuring that normal conditions have been established and maintained.

We would also point out, in conclusion, that our work has been simplified by the fact that the product we have been sizing has consisted almost entirely of one mineral; namely, pyrite. Single mineral sizing is much simpler than the sizing of mixed pulps, owing to the fact that each mineral in a particular jar has its own average size depending upon its specific gravity and consequently the percentage of each mineral present in the jar has to be determined, after the elutriation has been run, before accurate information regarding the fineness of the product can be obtained. We believe, however, that the type of material elutriated will not materially affect the procedure described in the foregoing pages other than that if products of low specific gravity are being elutriated it may be necessary to decrease the weight of the original sample taken for sizing, because of the relatively greater number of particles such samples contain.

APPENDIX 2.—QUANTITATIVE DETERMINATION OF PYRITE AND PYRRHOTITE IN NORANDA ORE AND MILL PRODUCTS*

WHEN analytical methods were developed for determining the percentage of pyrite, pyrrhotite and magnetite in massive sulfide ore and mill products, we had no idea of publishing them, and our chief reason for presenting them now is that we have had numerous requests asking that they appear in some permanent form. We would suggest, therefore, to potential users that the applicability of these methods to their problems will depend upon the degree of similarity in the mineralogical composition of their ore and that at Noranda. (See mineralogical analysis on page 570.)

The initial work connected with the development of these methods was carried out on pure minerals, which were tested in varying strengths of different acids to determine their relative solubility. As a result of these tests, it was found that pyrrhotite was readily soluble and pyrite practically insoluble, even after fine grinding, in a hot solution consisting of two volumes of water and one of hydrochloric acid.

Similar tests on the solubility of magnetite and chalcopyrite in hydrochloric acid solution of this strength showed that magnetite when present in amounts not exceeding 3 per cent might be considered as also being entirely soluble, and that though chalcopyrite was slightly soluble the percentage of this mineral that was dissolved did not necessitate a correction for it except in the analysis of copper-concentrate samples. Briefly, then, the determination of pyrrhotite depends upon its solubility and that of any accompanying magnetite in hot 2 to 1 hydrochloric acid, and the determination of pyrite on the decomposition in nitric acid of the filtered residue that remains from the hydrochloric acid treatment. The amount of iron that goes into solution in each of these acids can then be readily determined and the necessary corrections for magnetite and chalcopyrite dissolved in each can be made as directed in the detailed analytical procedure that follows.

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* In presenting this appendix, the author wishes to acknowledge, in particular, the work of R. J. Morton, of the concentrator metallurgical staff, who not only played a major part in the development of the procedures here described, but also rendered material assistance in preparing them for publication; also the part played in their initiation by B. Robinson, formerly of the concentrator staff, who first conceived and demonstrated the possibility of the quantitative determination of pyrite and pyrrhotite in Noranda ore by chemical methods.

METHOD OF PYRITE ANALYSIS

1. Weigh out into a 300-c.c. beaker 1-gram sample of ground pulp if pyrite content is in excess of 10 per cent, or 2-gram sample if pyrite is less than 10 per cent.
2. Add 50 c.c. of 2:1 HCl (2 parts water to 1 part HCl by volume).
3. Heat on hot plate for about 10 min., till evolution of H_2S fumes is finished as denoted by testing with lead acetate paper.
4. Filter with suction pump through a Gooch crucible lined with an asbestos pad.
5. Wash thoroughly with hot water.
6. Transfer crucible with contents to a 500-c.c. beaker, add a pinch of potassium chlorate to residue in crucible, then add sufficient concentrated nitric acid to decompose sulfides.
7. Evaporate to dryness on hot plate, and bake for 25 minutes.
8. Add 50 c.c. dilute hydrochloric acid and wash down with water.
9. Heat until nearly boiling, then add stannous chloride drop by drop until solution becomes colorless.
10. Cool solution and dilute to 300-c.c. bulk.
11. Add 10 c.c. concentrated mercuric chloride solution.
12. Add 15 c.c. titrating solution and three drops of indicating solution.
13. Titrate with potassium dichromate solution to a permanent violet color.

Notes.—

a. If considerable copper is present the reduction of the HCl solution must be done by boiling with test lead.

b. Alternative to step 7: Evaporate until nearly dry. Add 10 c.c. concentrated sulfuric acid. Take to white fumes denoting removal of all nitric acid.

c. If alternative to step 7 is used, substitute following for step 8: Cool and take up with 50 c.c. dilute hydrochloric acid. Wash down with water.

Solutions Required:

(1) *Standard potassium dichromate solution:*

1 c.c. = 0.00466 gram Fe

4.09 grams per liter

1 c.c. = 1.0 per cent FeS_2 in 1 gram original sample

(2) *Stannous chloride solution:* Dissolve 150 grams stannous chloride in 250 c.c. concentrated hydrochloric acid and then add 500 c.c. of water.

(3) *Mercuric chloride solution:* Saturated solution of mercuric chloride required (60 to 100 grams per liter).

(4) *Titrating solution:* 150 c.c. sulfuric acid, 150 c.c. phosphoric acid and 700 c.c. water.

(5) *Indicating solution:* 1 gram di-phenylamine dissolved in 100 c.c. concentrated sulfuric acid.

For rapid work it is essential that a suction pump and a Gooch crucible be used for filtering. Also, it is advisable to use the alternatives to steps 7 and 8. The use of di-phenylamine as an internal indicator was decided upon because of the necessity for speed, and its use has been entirely satisfactory. Application of the correction for copper in the determination of pyrite is as follows. A rapid determination of the copper in the sample is made by the fluoride-iodide method.[8] As in all samples dealt with the copper is present in the form of chalcopyrite, the percentage of iron in the chalcopyrite can be calculated from the chemical composition (Cu, 34.5 per cent; Fe, 30.5 per cent; S, 35.0 per cent) as follows:

$$\text{per cent Fe} = \frac{\text{per cent Cu} \times 305}{345}$$

The figure so obtained is then multiplied by the factor for pyrite, which is 2.146, and the product is deducted from the original titration figure for a 1-gram sample and the balance that remains represents the percentage of pyrite in the sample.

METHOD OF PYRRHOTITE ANALYSIS

The method for pyrite analysis is followed up to and including step 4; the filtered solution from this step is then reduced by stannous chloride, and the iron content of the solution determined by following the same procedure as in the pyrite method. Using the standard dichromate solution given above, for determining the percentage of pyrite directly, it is necessary to multiply the titration reading by 0.750 to obtain the percentage of pyrrhotite as $Fe_{12}S_{13}$ in a 1-gram sample. The formula for pyrrhotite given here naturally applies to Noranda ore and should not be used for another ore without being checked.

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[8] H. N. Thomson: Copper Determination by the Fluoride-Iodide Method. *Eng. & Min. Jnl.* (1932) **133**, 278.

METHOD OF MAGNETITE ANALYSIS

1. Weigh out into a 300-c.c. beaker 1-gram sample.
2. Digest thoroughly with bromine and concentrated nitric acid on hot plate until no further reaction takes place.
3. Dilute with large quantities of water by successive decantations until all the iron in the solution is removed.
4. Add 50-c.c. 1:1 HCl. Warm until the magnetite goes into solution.
5. Reduce the solution with stannous chloride and determine the iron content, following the same procedure as in the pyrite method.
6. Calculate the percentage of magnetite as Fe_3O_4.

The application of the correction for magnetite to the method for the determination of pyrrhotite is as follows: The percentage of iron soluble in hydrochloric acid is determined by the pyrrhotite method. The percentage of iron in the form of magnetite is then determined on a duplicate sample using the above method. Subtraction of this figure from that obtained by the pyrrhotite method will give the corrected percentage of iron as pyrrhotite in the sample. This figure is now multiplied by the factor 1.622 and the result will be the percentage of pyrrhotite as $Fe_{12}S_{13}$.

A Review of Black Hills Metallurgy

WITH PARTICULAR REFERENCE TO THE HOMESTAKE ORES

BY ALLAN J. CLARK,* MEMBER A.I.M.E.

IT may be said that the history of gold in the Black Hills begins with one of South Dakota's most interesting relics, the so-called Thoen Stone, now in the Adams Memorial Hall at Deadwood. This slab of reddish sandstone, about 14 in. square, was found in 1887 by Louis and Ivan Thoen, at the base of Lookout Mountain, near Spearfish, a town in the northern foothills of the Black Hills uplift. Both sides of the stone bear clearly legible inscriptions. One side reads:

> Came to these hills in 1833. Seven of us, De Lacompt, Ezra Kind, G. W. Wood, T. Brown, R. Kent, Wm. King, Indian Crow. All ded but me, Ezra Kind. Killed by Indians beyond the high hills. Got our gold June 1834.
>
> On the reverse side:
>
> Got all the gold we could carry. Our ponys all got by the Indians. I have lost my gun and nothing to eat, and Indians hunting me.

Attempts were made to trace these men. Relatives of Brown were found in Missouri, and it was learned that he had left for the West in 1832, with one Kent. Kind's relatives were also found, and stated that he had started West the same year. None of them had been heard from since their departure.

So Kind failed to get out with "all the gold we could carry"; 15 years later California became the center of the first great gold rush, and the Hills waited 40 years for their gold to be rediscovered. It is interesting to speculate on what might have been "the course of Empire" had this little group won through. The history of Dakota, of Montana, indeed, of all the Northwest, might have been very different.

When, late in 1875, the goldseekers arrived, they passed quickly northward from the first discovery near Custer, and soon were established on Deadwood Creek, along which, as well as for short distances above the mouths of its tributary streams, Blacktail and Bobtail, placer operations went forward vigorously.

These placers were near the northern outcrop of the Homestake orebody. Their gold was partly derived from this, perhaps in greater quantity from the erosion of the conglomerate or cement deposits, of Potsdam time, themselves a "fossil placer" or beach deposit formed by

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Manuscript received at the office of the Institute July 23, 1934.

* Metallurgist, Homestake Mining Co., Lead, South Dakota.

erosion of the Homestake ledge by the sea which surrounded it in geologic time.(1)*

In a general way, these placers ran from Golden Gate, where the Father de Smet mine, most northerly of the Homestake group, approaches Deadwood Creek, through Deadwood, some three miles below. The richest section is said to have been immediately below the junction of Blacktail and Deadwood creeks.

It is probable, too, that these cement deposits were the first to be mined and milled, although this is not clear, since in the years '76, '77 and '78 a large number of small custom mills of from 5 to 25 stamps were put in operation, both along Deadwood Creek and in Lead.

Early Mills

The Homestake was located by the Manuel brothers on April 9, 1876. They had previously acquired an interest in the Golden Terra, and in his reminiscences, Moses Manuel mentions having sold the Father de Smet lode claim. The Manuels milled the first Homestake ore during the winter of 1876–77, in an arrastre which they built at Kirk. They took out $5000. In the spring of 1877 they built a 10-stamp mill, and in the fall of that year sold the claim to Haggin, Hearst and Tevis, who were the principal owners of the Homestake Mining Co. at its incorporation, Nov. 5, 1877.

At about this time all the other companies since merged with the Homestake also came into existence—the Highland, next to Homestake on the north, then, still proceeding northward, the Deadwood Terra (an earlier consolidation of the Deadwood and Golden Terra), then the Caledonia, which retained its individuality longer than the others, but eventually passed, with them, to Homestake ownership.

The first Homestake mill, of 80 stamps, was built by the Union Iron Works of San Francisco, and started crushing ore July 12, 1878. Six months later work began on the Golden Star mill, of 120 stamps, this going into service Sept. 1, 1879.

Yates, in a historical review of the mine, written for the fiftieth anniversary in 1926, notes that the first annual report of the superintendent stated the reason for this second mill to be that "as there was a large amount of low-grade ore profitable to work with larger milling capacity, the trustees deemed it advisable to erect a new 120-stamp mill," thus giving early recognition to the importance of large tonnage to low-grade operations.

The Father de Smet mill, of 100 stamps, was built in 1878. In 1879 the Deadwood mill of 80 stamps, and the Caledonia mill of 60 stamps were built, and in 1880, the Highland mill of 120 stamps and the Golden

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* References are to the bibliography at the end of the paper.

Terra mill of 80 stamps completed the group.⁽²⁾ Thus the first phase of Black Hills metallurgy was in full swing by the latter date. Amalgamation was almost the only means of recovery, for concentration contributed little to the total. The stamps all weighed about 850 pounds.[1]

Hofman⁽³⁾ is our best authority for the earlier years of this period; in the next decade Rickard⁽⁴⁾ is the most satisfactory reference, and in his "Stamp Milling of Gold Ores" has given one of the most readable technical books ever written, although some of his strictures upon the methods then pursued at Homestake were, in my opinion, unwarranted, perhaps reflecting the impressions gathered from a hurried visit rather than a judgment based on a full knowledge of underlying conditions.

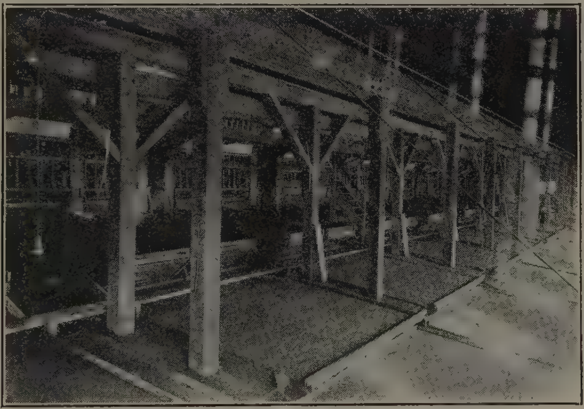

FIG. 1.—HOMESTAKE MILL IN 1898.

One of the early authorities states that the first mills were brought from Colorado. The reference is probably to small custom mills set up in Central City, for the mills of the Homestake Company were from California, and the Highland was by Fraser and Chalmers. (Fig. 1.)

Thus Homestake set out upon its milling way with the rather narrow and shallow mortar of California practice, instead of the wider and deeper one that typified Colorado design. It may have been a fortunate chance; it may have been deliberate choice. We do not know. But we do know that the management was alive to the necessity of maintaining a large tonnage, and Yates, in his examination of old reports of the Homestake,

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[1] In later years, these mills all came into possession of the Homestake Company, and were operated by it, but under new names. For the sake of reference, these changes are recorded here:

OLD NAME	NEW NAME	PRESENT STATUS
Father de Smet	Mineral Point	Dismantled
Deadwood-Terra	Pocahontas	Operating
Caledonia	Monroe	Dismantled
Highland	Amicus	Operating

notes that the stamp duty in 1882 was but 2.5 tons, whereas it had reached 4.0 tons by 1888. Hofman devotes his paper to a discussion of the variations in mill design, stamp design, mortar design and amalgamation details.

Design of Mills

As to design: Homestake, Golden Star and Highland had bins in center, and stamps back to back; Deadwood and Golden Terra, after consolidation, were similarly built; Caledonia's stamps were all in a single line, with issue in one direction, and Father de Smet had two rows of bins, next to the outer walls, with stamps in two rows, facing one another, and amalgamation plates sloping in either direction from these to a tailing launder through the center of the mill.

The writer has seen all these mills in operation, and has had personal charge of their operation at one time or another. In his opinion, the first three mentioned were the better design, with the Highland perhaps the best of the lot. The other three mills were idle for many years, so suffered from lack of the careful upkeep given the Lead group. But the Father de Smet was hopeless. It was argued that the design brought all the plates into the open, so that they could always be under surveillance; unhappily, the bins cut off so much light that the room was in semidarkness. Hofman noted this. Also, the plate arrangement was absolutely inflexible, for, since the two lines of plates met at the central tailing launder, extensions and additions within the mill were impracticable.

Much importance attached to mortar design, since this influenced amalgamation. Caledonia retained a wider mortar, used two inside plates, dropped 12 in. instead of 9, and at a correspondingly slower rate. Its stamp duty was 25 per cent less. Better amalgamation was claimed. Examining the data, it appears that the larger inside plate area was gained by a reduction in discharge area, which in turn necessitated a coarser screen. One concludes that Caledonia may have amalgamated more of the gold it liberated by crushing, but that, since it discharged its tailing at a coarser size, it was liberating a smaller percentage than were the Homestake mills.

If the several steps are considered critically, it appears that:

1. Caledonia paid too high a price for inside amalgamation.

2. The use of narrowed sluice plates was absurd. The millmen may not have been aware of it, but there was another reason for feeding quicksilver to the battery, besides the necessity of such addition to accomplish "inside amalgamation." This was to prepare the finer particles of gold—those in the slime and in the 200-mesh sand—for subsequent recovery on plates.

Naturally, to make these small particles adhere, there had to be an opportunity for contact, which was not provided in the narrow sluice boxes.

3. Similarly, these fine particles had little opportunity to come to rest, or into contact with larger particles of amalgam, in the sumps. The sumps served to reclaim dry particles of amalgam broken from the chuck blocks within the mortar or loosened from the apron plates, and those in which attached pyrite prevented adherence to the plate. Also they served to recover loose quicksilver.

The sump was a necessary safety provision in the circuit, but in my opinion, the attitude generally assumed, of satisfaction with overly large sump recoveries, was wrong. It was intended that recovery should be made in the equipment ahead of the sumps. An excessive recovery in the latter should have prompted critical examination of chuck-blocks and apron plates.

CONCENTRATION

Although never of major consequence, concentration, in several forms, was attempted during these years. Caledonia used a 4-ft. length of blanket below the apron plates, and followed these with quicksilver traps, one of which was provided for each plate. Blanket concentrates, after amalgamation in pans, were said to run $90 a ton. Homestake followed this practice for a time, although it introduced changes of doubtful value. It used a larger area of "blanket," and substituted Brussels carpet for the lighter blankets of Caledonia. Naturally, the concentrates were of lower grade—it is a safe inference that the heavy carpets discouraged handling and that washing was not adequate. Be this as it may, we are told that "being too expensive, it was given up," presumably about 1886.

After discarding blankets, Homestake installed "bumping tables"; the "Perfection" table usual in Colorado practice. Rickard considered the equipment inadequate, but these, too, were abandoned after a time, and it was not until the middle nineties that jigs were used to produce a considerable tonnage.

Again, surveying concentration during these years from the standpoint of present knowledge, we may infer:

1. That Caledonia did a better job. Presumably, it skimmed the cream of the sulfides, keeping tonnage low and recovering little but the rich pyritic material.

2. Homestake, installing a separate blanket house, either overdid the extent of blankets, or gave them insufficient attention. Either they recovered low-grade pyrrhotite with the richer pyrite, or they contaminated the pyrite with gangue minerals. This was true of the "bumper" product, as well.

Hofman cites $90 as Caledonia concentration; in the nineties, when Homestake began to produce in quantity, the grade was rarely over $8. The answer was the same—pyrrhotite.

In the early days, there was little incentive to make concentrates unless the amalgamable gold won by re-treating them would pay for the operation. There was no immediate market, and no suitable process was available. To have stored for future use might have appealed—it probably would be done today. But the first generation of American miners was not worrying about the future. It was living in the present, exploiting a new country. These miners were resourceful; their judgment in matters of mining and milling was acute, but conservation did not interest them. And from the economic standpoint, it is doubtful that storage of concentrates would have paid. Such concentrates would not have been amenable to cyanidation, since a month's exposure would have generated more cyanicides than the ore could support; fine grinding and re-amalgamation would have been doubtful, since the sulfides were already fine, and should have yielded most of their free gold. Only with selective flotation, about 1930, could there have been a chance—and here, again, the oxidation products might have given some trouble.

The Deadwood and Delaware smelter, built in 1891, furnished a market for concentrates. The "jig-house," treating tailing of the three Homestake mills at Lead, was the answer, and it operated until cyanide treatment of the sands proved a more profitable operation.

Early mining practice, through inadequate sorting, long exercised a detrimental influence upon milling, Hofman says:

> As the gold is finely disseminated throughout the entire vein-matter, comparatively little sorting in the mine can be expected . . . when the Nevada system of timbering was in use no distinction was made between mill-rock and waste, but it was considered that the cheapest way to get rid of the latter was to run it through the mill. Lately, less waste is sent to the mill, but large quantities are still got rid of the old way.

He also describes the method of "sampling" for free gold, by panning, and comments: "as these are the only determinations made it can be readily seen that the amount of non-free-milling gold which enters the mill is not known." Rickard notes:

> There is no marked definition between what is valuable, and therefore ore, and what is valueless and therefore country rock. The pay ore usually carries sulfides . . . The ore is broken without any attempt at sorting. Its low tenor and the mode of its occurrence alike render such a step impracticable. At one time the porphyry, flanking and overtopping the ore-bodies, was sent to the mills.

These comments indicate how natural the procedure was, particularly in the earlier years. Yet it was probably the great error of the period, and the weak spot, in my judgment, was in the inaccuracy, the undependability of the "sampling." I am not referring to the taking of the sampling underground, although a systematic procedure of this nature was the first step essential to remedying the condition. I refer to the panning, the estimating of free gold in the samples submitted to the "sampler."

When a fair amount of gold was present, no unfortunate consequences would follow an error, but when the sample was waste or of marginal value, the valuation became a blind guess, based presumably on the amount of sulfides present, and the barren rock of the walls often made a fair showing of these. Hofman detected the weakness of the method, but he missed the vital point. It was not lack of knowledge of the amount of "non-free-milling" gold that mattered; it was lack of knowledge of the non-gold-bearing rock that entered the mill. The net effect was that 25 per cent, possibly more, of the stamps were dropping on waste. Grade of ore and of tailing was reduced in proportion.

About 1890, treatment of the Potsdam ores began to develop.(5) And during the following decade Homestake entered the last phase of the all-amalgamation era.

AMALGAMATION

We note the failure of the Ball steam stamp in 1889, obviously because it could not discharge, through the available screen apertures, the amount of ore it crushed. It is apparent that such a machine was altogether unsuited to crushing through a No. 7 slot screen, which would be rated as equivalent to 30 mesh, and, in fact, with the 850-lb. stamps, would discharge a product of which 75 per cent was finer than 100 mesh. Ore and wood scrap banked against the screens and broke them. Had the stamp not failed from this cause it would have failed as an amalgamator, because of the brief period of contact of ore and quicksilver within the mortar.

We find the Homestake mills, in the middle nineties, at about their best as amalgamating machines. The screen has become slightly finer—No. 8 slot—this being possible because of frequent whipping to dislodge the chips of wood constantly tending to choke the apertures. The stamp duty was almost 4.25 tons, and the narrow sluice plates had been replaced by three additional rows, each 12 ft. long and of increasing width as they receded from the mortar and apron plate.

These plates were of silvered copper. The amalgam was kept much softer, and the catch, approximately 40¢ a ton, was mainly from the finer particles of gold, 100 mesh or finer.

It is recorded that in the earlier days it was customary to warm the mill water; now cold water is in demand, temperature below 50° being desired. The softer amalgam, higher in quicksilver content, seemingly became dissociated from part of its quicksilver at higher temperatures. This ran down the table to the traps, the plate, meanwhile, appearing almost bare, but, as a matter of fact, retaining the gold in a harder scale beneath the beads of quicksilver which covered the table. The appearance of a table under these conditions was alarming, although I was never able to find that any loss was incurred.

The influence of inside amalgamation on the catch of these secondary rows of plates may be noted. When it was dispensed with, the coarser gold would stop somewhere down the tables, but the very fine particles would not. Assay of the minus 200-mesh fraction of tailing would invariably be higher when a battery was run, for experimental purposes, without inside amalgamation.

Reviewing battery practice of this time, one is impressed by the balance between crushing and recovery. In the later days, when cyanidation was added to amalgamation, we made numerous tests to increase the stamp duty. Naturally, every such change involved a shorter contact with quicksilver within the mortar, and consequently a higher slime-tailing assay. Not until all the slime was brought to the treatment plant built in 1907 at Deadwood, were we able to drive the stamps beyond the capacity established in the nineties.

The Potsdam, or siliceous ores, centering in the region about Terry's Peak, were first located(1,5,6) in 1877. In 1879 an arrastre was run, unsuccessfully, upon ores from the Empire mine. In 1880, an unsuccessful attempt was made at the Portland mine to treat the ores by pan amalgamation, which was followed by unproductive experimentation with other methods. In 1883 the Welcome Company passed through a similar history of unsatisfactory metallurgical experimentation.

The Buxton Mining Co. built a mill in 1886, and experiments were made there, by Ankeny, in which bromine was employed. All commentators agree that this attempt should have succeeded; that it stopped just short of success, and that, had it been attempted a few years later when more suitable equipment was available, it would have succeeded. From the perspective of nearly half a century, one may feel some skepticism. It is difficult to see that bromine had any virtues not shared by chlorine, and the latter was cheaper and probably less obnoxious to handle. At all events, time has sustained this point of view. Whatever the cause, the attempt was abandoned, and the property passed, some 15 years later, to the firm of Lundberg, Dorr and Wilson.

About 1889, experimental work with chlorine, apparently a crude attempt at the Plattner process, leaching the ore in covered vats, was made at Garden City. Also, at about the same time, the State School of Mines demonstrated that the Plattner process was successful on such ores as were low in silver content.

In 1891 a small plant was built at Rapid City for the treatment of ore from the Gilt Edge, near Galena, and in the same year the Golden Reward Co. erected a 50-ton plant in Deadwood, using the barrel process.

This plant commenced operations in 1891, and was later enlarged to a capacity of 100 tons a day for blue ores, and 150 tons for red ores. The ore was put through a crusher, then dried. The dry ore went to fine and rolls, which reduced it to 8 to 12 mesh. It was next roasted in Howell-White cylinders (red ores) or in Bruckner cylinders (blue ores), cooled and elevated to bins whence it was drawn through hoppers into the chlorination barrels.

The barrels were of iron, lead lined, and had a capacity of about five tons. About four tons of ore, 12 lb. of bleach, 18 to 20 lb. of sulfuric acid per ton constituted a charge. The barrels were slowly revolved for 1½ hr., water being admitted under pressure after chlorination was complete to leach the chlorinated ore through a filter contained in the barrel.

The gold-bearing solution was treated with hydrosulfuric acid gas, in some cases after a preliminary treatment with sulfurous acid to reduce chlorine. The sulfides were filter-pressed, dried, roasted in muffles and melted to bullion.

The Kildonan mill, 100 tons capacity, was in operation in 1895, the process being essentially the same as Golden Reward.

Smith, from whose paper I have drawn freely, states that(5) "the saving of precious metals is not so good as might be desired." Langguth states that extractions were from 90 to 97 per cent on heads that ranged from $12 to $20. Chance(6) states that costs ranged from $3.50 to $4.50, and complains of what he considered the excessive treatment charge of $9.00, levied alike by chlorination mills and smelter. Side lights on this question are found in mining journals of the time. Thus, Alaska-Treadwell, chlorinating a concentrate much heavier in sulfur than the Black Hills ores, reported costs of $7.82, about equally divided between labor and materials. A statement in the *Engineering and Mining Journal* (Oct. 30, 1897), indicates that Golden Reward's recovery was at the rate of $25 a ton. The tailing of the Rapid City plant, which was treated later by cyanidation, was stated to run from four to five dollars. Up to the time of the chlorination plants and smelter, ores of less than $30 value could not be handled at any profit.

SMELTING

During 1889 Franklin R. Carpenter,(7) then head of the State School of Mines, began experiments in pyritic smelting, basing his work largely upon the methods employed at Kongsberg, Norway. After the usual course of experimentation, the Deadwood and Delaware smelter was built at Deadwood. This plant operated until 1904, and was, next to the Homestake, the most important producer during this period. He described the process several times. Dankwardt, a later superintendent, also described it.(19)

In its beginning, the ore was smelted with dolomitic limestone and pyrite. Slags approximated 50 per cent silica. Later, copper ores were substituted for part of the pyrite. Dankwardt gives the daily charge as: ore, 180 tons; copper ore (5 per cent copper), 60; pyrite, 100; limestone, 180.

One of the major difficulties was to find a suitable source of pyrite. The Homestake concentrates, although very fine, were the most satisfactory, but made much flue dust, and eventually it was customary to pre-smelt them in large reverberatory furnaces, adding the low-grade iron matte produced to the ore charge. The use of these concentrates was mutually advantageous. Had this outlet not been available, there would have been no incentive for Homestake to produce them. When Homestake began cyanidation of sands and discontinued its concentrator, the smelter was compelled to replace them with graphitic pyrite, barren in gold, and the unsuitable nature of this was probably one of the causes of the eventual passing of the plant.

One of the characteristics of the operation was the formation of iron sows, sometimes attaining a weight of 20 to 40 tons, and carrying some 20 oz. of gold per ton. These were first sold, then treated by placing in a reverberatory hearth when access was permitted by reason of the top flue being off for repairs. Pyrite and copper ores were placed over it, and, with three days of firing, the sow was cleaned up. Carpenter favored the formation of these.

Dankwardt, later, claimed to have obviated the necessity of forming them, partly by the freer use of copper ores; partly by widening of the furnace at the tuyeres. Normally, about 5 per cent of matte was made.

Chance states that costs were from $4.75 to $5.75. He deplored the treatment charge of $9 levied on outside ores, suggesting that such charges made about $15 the low limit of ore that could be mined at a profit. Doubtless, as he hinted, these high charges would compel, and were intended to compel, the passage of mining claims into fewer and stronger hands. Certainly, the first companies to turn to cyaniding were some of the smaller and financially weaker properties.

The withdrawal of Homestake concentrates was but the first of a series of unfavorable conditions. Decreased production of high-grade ore, together with the facilities for treatment of medium-grade ore by cyanidation, played their part, and labor difficulties contributed to its closing about the middle of 1903. For two years thereafter it was held in readiness to reopen, but favorable conditions did not return.

Cyanidation

Cyanidation began in the Hills in 1892, at the small and poorly equipped plant variously known as the Golden Gate, the Rossiter, or the First Ward mill. This treated sand from the oxidized siliceous ores.

It was a dry crushing plant, which crushed with rolls and treated the unclassified ore in vats. Its priority is about all that merits recollection, and its operation must have been of doubtful profit since it appears to have run at but about half its rated capacity, and to have inspired few imitators, for some years passed before further progress was made with the process.

In 1896, according to Fulton,(9) the Rossiter plant treated 6500 tons of oxidized siliceous ore, as against 75,000 tons treated by chlorination. There was little to suggest that any great metallurgical change was imminent.

From 1896 to 1898, a few small plants, of the crudest description, were built, but it was not until 1899 that the real swing to the process began. Homestake's pilot plant may have stimulated innovations; at all events, development thereafter was rapid, so that by 1901, cyanidation was used for 88,000 tons and in 1904 no less than 550,000 tons were treated in 16 mills operating on Potsdam ores, with two more in process of construction. In addition, Homestake's two sand plants were in full operation, its experimental work on slime had been successful, and construction of that mill began during the following year.

Those were the golden years of Black Hills metallurgy, and the work of the metallurgists of that time has left its imprint on cyanidation, and indeed, upon hydrometallurgy in general, throughout the world.

The first application of crushing in solution to be made in this country was by Henton in 1899, although this had been done in New Zealand two years before. Randall had the idea of countercurrent decantation, failing to develop it because he lacked suitable equipment. It remained for Dorr(11) to develop it after his thickener had provided the means. Dorr also developed his classifier at the historic Lundberg, Dorr and Wilson mill on the Buxton mine near Terry, and here he made the first really successful application of the Moore filter.

Meanwhile, at Homestake, Merrill(8) brought single-filling sand treatment to a high degree of perfection, made a going method of zinc-dust precipitation and developed the sluicing filter press which bears his name.

Cyanidation of the Potsdam ores followed two general lines. Some—generally those not requiring fine grinding—were dry crushing plants, rolls being favored for the final size reduction. The crushed ore, with no attempt whatever at classification, was leached in vats; the collected dust, up to 7 per cent of the total, was treated separately, usually by agitation and decantation.

Ores requiring finer crushing were crushed in solution, usually in the stamp battery, although the Lundberg, Dorr and Wilson mill used a 6-ft. Chilean mill of the Monadnock type. Classification was generally effected in cones, the sand was leached in vats, the slime treated by agitation and decantation. Dorr developed the classifier when other means were inadequate to free his sand from slime, and followed with the Moore filter to recover dissolved values from his slimes. From the perspective of 25 years the Lundberg, Dorr and Wilson was the outstanding mill of the latter class, although not of greatest capacity. Similarly, although the Imperial mill was the largest and best appointed of the former class, Wasp No. 2 became the foremost producer in the former class, profiting by the ready extraction from 8-mesh material to establish phenomenally low mining and milling costs.

By 1906 the battle of the processes ended with the triumph of wet crushing. In this year Horseshoe converted its old Kildonan mill to replace its large wet-crushing mill at Terry, which had been destroyed by fire in the previous year. Golden Reward also converted its mill during this year. Wasp by 1911 was treating $2.20 material at a profit. In the *Engineering and Mining Journal* in 1907, Dorr commented that four-fifths of the ore then being treated could not have been handled at a profit with the milling costs of five years before.

Unfortunately, mixed with the oxidized ores were the unoxidized, or so-called blue ores, which to this day remain the *bête noir* of Black Hills metallurgy. Sometimes occurring in masses that may be segregated from the oxidized or red ore, they more often are in close admixture with the latter, so that separation is practically impossible. As a result, much of this ore went to the mills with the red ore, extraction varying with the relative proportions of the two. As the grade of mill feed decreased, the margin of the profit became less—a drop of a very small percentage in extraction was enough to erase it—and so this type of ore, of a grade from $5 to $7, accumulated in the mines.

The peak of production from these Potsdam ores was reached, so far as I am able to ascertain, in 1905, although that of the following year was almost as great. Labor trouble intervened in 1907, and again in 1910. The large Horseshoe mill burned in 1905, the first Wasp No. 2 in 1909, and in 1912 the first Mogul mill at Pluma was destroyed. By 1911 but five mills remained; in 1917 there were but three, in 1918 but two, and from 1919 the Trojan was the only survivor. The war boom, with its great rise in the prices of commodities, was too great a burden. The narrow-gage railroads that served these mines were abandoned and the rails withdrawn. Today the problem for the regeneration of these mines is as much one of transportation as of metallurgy. Both could be developed; neither could be expected to be available at such low costs as formerly.

Cyanidation at Homestake was initiated in 1899, that summer and the one following being devoted to the operation of a 60-ton pilot plant for the treatment of sand from the mill tailing. This experimental work was directed by Merrill, who designed and operated the two sand plants and later, the slime plant. All these mills are still operating, and until this year they constituted all the Homestake's cyanidation plant.

The pilot plant employed jigs for the removal of slime. Great emphasis was placed on clean classification. It was learned very early in the work that frequent aerations were necessary to maintain a supply of oxygen in the solutions, and it was at this plant that the system of introducing compressed air beneath the canvas filter bottom was evolved.

SAND TAILING

In 1901 the No. 1 sand plant, for the treatment of sand tailing from the Lead mills, went into commission. This plant was situated down the gulch, about ½ mile below the mills, so that tailing could flow through the classification system and to the leaching vats by gravity. Classification was by jigs, similar to those used in the pilot plant and in the concentrator house, but within a year these had been superseded by a system of cones, in series, the third and last series being provided with an upward stream of water near the discharge, for a final washing of the last particles from the leachable sand.

Lime was not added until classification was complete, since, unfortunately, the local water supply was of a bicarbonate type, and reacted with the lime to precipitate carbonates, some of the slime, derived from the iron-magnesia carbonates of the ore, entering into this reaction. The effect was to precipitate a hard, stony scale upon the amalgam plates, and to almost inhibit amalgamation. Thus it was not possible to take advantage of the action of the lime to hasten settling, an action which in South Africa was held to favor amalgamation of the finest particles of gold.

This reaction must always be kept in mind, and streams of new water kept from mixing with alkaline pulp or water except where the resulting scale formation can cause no damage. Thus, launders, which may be scaled at intervals, are generally favored over pipes where such conditions obtain.

During 1901 and 1902 about 460,000 tons per annum were leached and about $540,000 recovered. About 40 per cent of this was plus 100 mesh; about 30 per cent minus 200 mesh. Leaching rates, as determined by measuring the fall of solution level on a tank during draining, was about 3½ in. an hour. Extraction was about 70 per cent and consumption of potassium cyanide was about two-thirds of a pound per ton of sand.

The first advance in method was, as already stated, during 1902, when cones replaced jigs in classification. In 1903 a period of aeration, preceding addition of cyanide, was tried. By this means consumption of cyanide was reduced to about 0.45 lb., and this preliminary aeration became part of the standard method. Shortly thereafter a fourth battery of cones was added to the classification system, with the effect of adding

100 tons of fine sand to the daily tonnage. With these changes made, methods continued with few further innovations until the middle of the year 1909, when sodium cyanide superseded potassium cyanide. During these years consumption ranged from 0.38 lb. up to 0.58 lb.; extractions remained almost unchanged, tonnage rose to 640,000 in 1906 and in that year a recovery of $711,000 was made. At the same time, the vats were deepened, the working strength of cyanide reduced, the leaching rates slowed to about 2 inches.

Following the successful operation of the Lead sand plant, a similar plant was built at Blacktail, on Deadwood Creek, to treat tailing from the mills on the northern end of the ledge—the Deadwood Terra, Monroe (formerly Caledonia) and Mineral Point (formerly Father de Smet). (Fig. 2.)

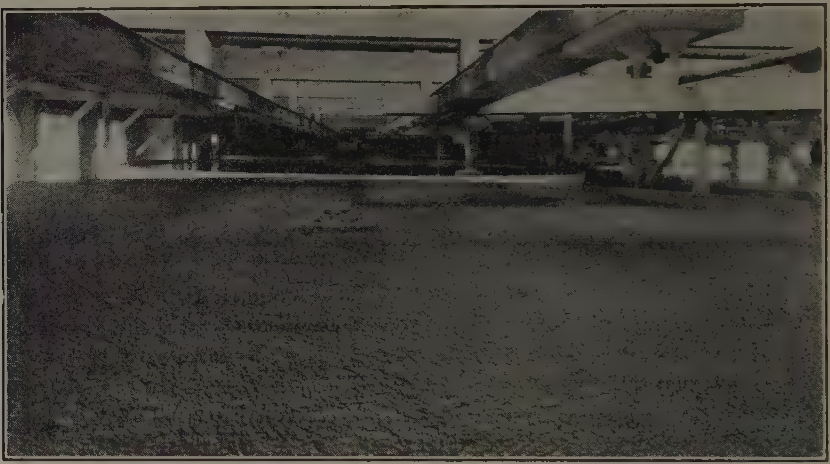

FIG. 2.—CYANIDE SAND PLANT AT HOMESTAKE IN 1905.

This plant treated tailing from a more oxidized and poorer ore than that at Lead. The sand treated was rather coarser, ranging up to 44 per cent plus 100 mesh, yet extractions were better, 75 per cent being about the average and 72 per cent the minimum. Sand assaying $0.68 has been treated at a profit.

With the sand plants completed, Merrill turned his attention to the slime, then averaging about $1.00 to $1.10 a ton. Through 1905 and 1906 he experimented in a small way, and in the latter year erected the plant at Deadwood, at the confluence of the streams draining the Lead and Terraville watersheds, to which slime from all the mills could flow by gravity. The method was by pressure leaching in filter presses of the plate and distance frame type, distinguished, however, by being equipped with a sluicing mechanism and discharging channels and ports which permitted the discharge of a finished charge without opening the press.

The presses, treating 25 tons at a charge, were some four times greater in capacity than any presses used before that date.

Lending themselves to the Homestake requirement of aeration both before and at intervals during the treatment cycle, they were also admirably designed to take full advantage of the ready permeability of slime from the unoxidized ore, which constitutes by far the major amount of Homestake ore. Parenthetically, it may be noted here that when oxidized ore, more colloidal in character, is treated, the press is by no means so successful, since the rigidity of design requires that a cake of full thickness must be treated, irrespective of its permeability.

The slime plant treated 253,000 tons ($169,000) in 1907, then for three successive years the tonnage advanced to about 550,000 tons ($445,000), and in 1911, 617,000 tons were treated. The maximum tonnage at this plant was recorded in 1914, when 692,000 tons were treated to recover $625,000. However, although this tonnage has never been surpassed, the output in 1933, at the old rate of $20.67 an ounce, was more than double that of 1914. About five years elapsed before slime tonnage reached 600,000 tons.

W. J. Sharwood and the present author(12) described at great length the metallurgical operations of this time, which may be said to have marked the high point of the first great period of growth. Since it is not the aim of this article to discuss details of treatment exhaustively, reference is made to this paper, where they are available.

At about this time it has been demonstrated that all the slime, even that of the lowest grade ore, was treatable. It had been recognized that some of the component minerals, particularly the hornblende particles, were almost barren, but they could not be removed at a profit, and fortunately the needlelike crystals of this mineral gave permeability to the whole mass, so that its presence was not altogether a waste.

This situation set the stage for further advances. With none of the mill tailing escaping cyanidation, maintenance of full amalgamation yield was no longer imperative. Tonnage per stamp might be increased, even though some fine gold escaped amalgamation; fine grinding might go forward on a larger scale than formerly, since it was now assured that the additional slime produced would go to cyanidation. Gradually, and as far as the design of the old mills permitted, these things were done.

SIZING

In 1906, the Denny brothers published a paper in the *Journal* of the South African Association of Engineers, which has had an important influence on all metallurgical advances since that time. Its advocacy of what has since become common practice—the sizing of samples under investigation, and the study of each grade—was at once appreciated at Homestake. Extended to further subdivide each size into concentrates and gangue minerals, and these in turn into amalgamable and non-amalgamable gold, it has determined all changes in Homestake flow sheets.

First applied to Homestake ores in 1906, it indicated that the coarser sizes might be reground at a profit, and further, that the sulfide gold was more readily extracted than was that of the gangue, although, because of inadequate means for making a clean separation, the full degree of this differentiation was not made apparent at that time. A modest plant, which withdrew coarse sand from the Lead tailing for grinding in Wheeler pans, was the first step in regrinding. Results checked the laboratory work, but the pans proved expensive to operate and adequate development had to await growth of slime treatment until all tailing, both sand and slime, could go to cyanidation. Thereafter pebble mills, four of 5 by 14 ft. and two of 5 by 18 ft., reground the coarser fraction of the Lead tailing.

These mills, individually, were in open circuit, but the plant as a whole was in closed circuit, the ground pulp being returned to the main tailing launder in advance of the classifying cones that withdrew the coarse sand for regrinding. In this manner sulfides were prevented from accumulating in the circuit.

Gradually, responding to these changes, the extraction at sand plants rose to 75 per cent by 1912, to 78 per cent two years later. By 1914, but 28 per cent of the sand charges was on 50 mesh, and 40 per cent was through 200 mesh. Recovery reached $840,000 by 1917. Gradually, too, the tonnage of slime increased.

During this period two major changes in accord with development in the cyaniding field were the installation of the Merrill-Crowe vacuum precipitation process, in 1917, and the use of low-strength "Aero" brand cyanide, in 1921.

During, and immediately after the war period, mining conditions had marked effect upon milling operations. In the first place the crude "panning" of samples had been discarded. Careful sampling had made it possible to keep much waste from the mills, with a corresponding increase in the grade of mill feed, but these benefits we deferred for some eight years, during which the "draw-hole" system of mining provided more than one-half the ore supply. This system, made necessary by conditions in the mine, has been described by Bjorge.(18)

Homestake South Mill

It was necessary, first, to mine the broken pillars left in the earlier days of mining, which could not be safely mined by ordinary methods, and, second, because scarcity of labor in the years of the post-war boom made this method necessary to maintain production. Finally, two of the old mills were carried on large pillars of good ore, which had to be mined at once, before the draw-hole system could be instituted. Accordingly, preparations were made to abandon the Homestake and Golden Star mills, which, it will be recalled, were the two original mills of the Homestake Company. In 1921 the South mill replaced them. This mill has been frequently described. Its main features and operating results may be briefly stated as follows:

Stamps were retained, but for fine crushing only. Weighted to 1550 lb. each, they crushed through screens with openings which in 1921 approximated ⅜ to ½ in. and, in the current year, approximate ⅞ in. Stamp duty ranged from 15 tons in 1921 to 22.5 tons at present. No quicksilver was fed to the battery, and, indeed, every effort is made to keep launders smooth and free from corners or ridges on which free gold might concentrate. The stamp product is separated into coarse and fine fractions by dewatering cones, which discharge to rod mills, 5 by 10 ft., using 3-in. dia. rods as grinding media. It is to these mills that quicksilver is added, the present quantity being about 0.25 oz. troy per ton. Some amalgam builds up in the mill, the scoop box, and the Dorr classifier, but the major recovery is in a recently patented amalgamator of local design. These are followed by two batteries of silvered plates, and these in turn by gravity cones, which withdraw the coarsest sand for regrinding in pebble mills. The total recovery in this mill by amalgamation is about 65 per cent, rather more than three-fourths of which is derived from the rod-mill amalgamation circuit. Operating costs have been more than $0.10 per ton below that of the surviving mills of the old type.

This period of draw-hole mining had no ill effects on cyanidation of sand, but it gave much trouble in slime treatment. Not only was the slime more truly colloidal, but it carried much organic matter, in varying amounts, the accumulation of 50 years.

Thickening equipment was inadequate. Slime treatment was slowed up through impermeable cakes in the treatment presses. Since the condition was known to be of a temporary nature, additions to neither were warranted, and it was necessary to waste some slime. By segregating the higher grade of slime from the lower levels of the mine, it was possible to waste only the fraction originating from draw holes, and only the finest fraction of this, in the overflow of the thickeners.

The organic matter was more difficult. Extractions slipped slowly from the 90 per cent usual before 1920, a minimum of 78 per cent being recorded in 1928. It was in the following year that some control was obtained over this by the addition of bleaching powder, generated at the plant by passing chlorine gas into quicklime, to the thick pulp before it entered the press. The requirement was considerable, and about $0.07 was added to the cost of treatment, so it was only available when conditions were particularly bad, or the grade of slime exceptionally high. By

1931 recovery had returned to 87 per cent, and since that date it has exceeded 90 per cent.

When the South mill stamps began to drop, four of the old mills were dismantled, the Amicus and Deadwood-Terra being the survivors. All the soil under the mortars, plates, and launders, and much of that elsewhere in the mill, was sent through the batteries, the ground nearest to the mortars often being excavated to a depth of several feet before all evidence of quicksilver disappeared. An effort was made to estimate the tonnage handled by each mill during its life, and these figures were compared with the recovery made from this final dismantling. In every case this approximated one-half cent per ton milled.

Modern Practice

About 1929 the draw-hole ore became of less consequence. The mine at once reflected this with a gratifying increase in the grade of mill feed. It soon became evident that the heavy sulfide ores of the deeper levels would present some difficulties in cyanidation. It was thought that selective flotation might offer an alternative means of treatment, but thorough testing disproved this, indicating rather a longer leaching with cyanide, together with as fine a grind as the grade of ore would warrant.

Although most of the gold is associated with the sulfides, there is so much disseminated through the non-sulfide minerals that the whole mill feed must be finely ground and cyanided. No fractional part can be selected for intensive treatment. Also, the sulfides extract readily, even at rather coarse size, whereas the gangue minerals even at 300 mesh will yield only about 80 per cent. Of the sulfides, pyrite is the chief carrier of gold, and the chief destroyer of cyanide; arsenopyrite rates next. Both of these yield their gold readily. Pyrrhotite, which is less rich in gold than the others, extracts less readily and less completely, but exercises little destructive action upon the cyanides.

No. 3 cyanide sand plant was built in 1933 (Fig. 3) to treat lower-level ore. This is segregated in part of the South mill bin, crushed there and passed through rod mills, whence it flows to the sand plant. A Dorr bowl classifier receives the pulp and delivers the plus 100 to plus 150-mesh fraction to two tube mills, 5 by 14 ft., using 2-in. balls as grinding media, and in closed circuit with a 6-ft. Dorr classifier. The mill discharge is equipped with amalgamators. Classifier discharge goes to Dorr bowl classifiers for separation into sand and slime, the separation being at about 350 mesh. The sand goes to vats, 44 ft. in diameter by 12 ft. in working depth, holding about 710 tons. There are eight of these, and the treatment cycle, exclusive of sluicing and filling, is about six days. Treatment follows the established Homestake routine, the high points of which are extremely low alkalinities, several periods of drainage and aeration, and precipitation of pregnant solutions with zinc dust. The latter step, however, is accomplished at the older plant, No. 1. Sluicing is automatic, water being introduced through the distributor and through nozzles about the discharge gates. A vat is completely discharged in about 4 hours.

It may be noted that, although the average size of tailing particles is less than it has been heretofore, the percentage of tailing leached as sand is not decreasing. Charges containing no plus 50-mesh and as much as 47 per cent minus 200-mesh material leach 2 in. per hour, and extract up to 90 per cent. Recent over-all monthly recoveries have reached as high as 95.8 per cent.

As this article is written, an addition to South mill is under construction. At its completion, all the milling operations will center here. In general, the procedure will be similar to that now current, but there are some modifications.

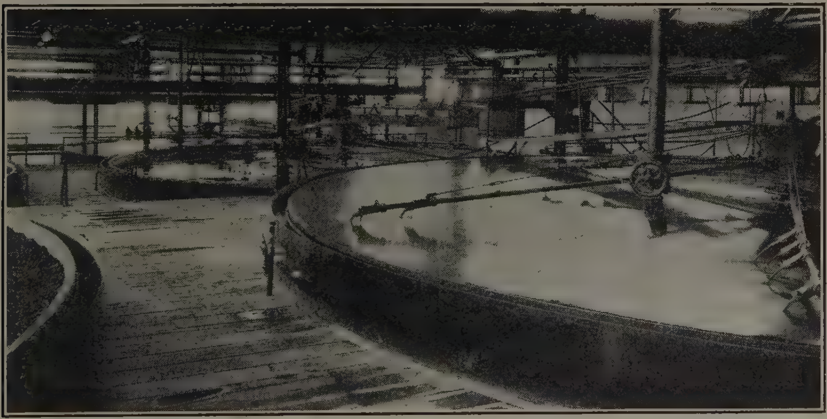

Fig. 3.—Cyanide sand plant at Homestake in 1934. Vat in foreground is filling.

Ore from the Ross shaft will come to the mill after having been crushed to about 1½ in., possibly slightly finer, in Symons crushers. It will be crushed by stamps weighing 1550 lb. and discharging through a screen with apertures 0.8 to 0.85 in. square. Thence to dewatering cones, the sand underflowing these being ground in Marcy rod mills 6 by 12 ft. in closed circuit with amalgamators and classifiers. Plates will not be included in the new set-up.

Classifier discharge will be delivered by bowl classifiers to Marcy tube mills, 5 by 14 ft. in closed circuit with amalgamators and classifiers. Steel balls, 1½ in. in diameter, are to be used for grinding. Classification will be in the existing system of cones at cyanide No. 1, where the sands will be leached. Slime will be treated at the slime plant.

Since 1933 a parting plant, using the Miller process, has prepared all doré bullion for marketing. Gold is shipped at approximately 994 fine, silver at 999.

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Milling Methods of the Porcupine District of Northern Ontario

By P. D. P. Hamilton,* Member A.I.M.E.

In any mining district where benefication of the ores is practiced, one can usually observe several types of flow sheets even though the various producers are milling ore from the same geological formations. It is obviously impossible to describe all the mills in the Porcupine district. However, the following description of some of them is of particular interest in that it shows where one mill has paid particular attention to corduroy blanket practice; one to gravity concentration of sulfides for intensive cyanidation of the values therein contained; one to flotation of values and cyanidation of this product only; and others where small variations of standard all-slime cyanidation practice prevail. In all cases it will be seen that cyanidation is the basic principle.

Discussions involving the difference in the flow sheets and the reasons for them will always be of value to those interested in the art. The author of this article, however, believes that such discussions should originate from the operators of the mills under discussion. For this reason no comparisons or discussion will be included in the description of the mills mentioned.

Should any reader of this article obtain information therefrom which will be of value to him in helping to solve his own particular problems, the author will consider that the article has satisfactorily served its purpose.

All values mentioned throughout this article are based on the value of gold at $20.67 per ounce.

Coniaurum Mines, Limited

The ore of the Coniaurium mines consists of a mineralized schist and quartz containing about 2.5 per cent sulfides. Owing to the low grade of ore developed in the mine, extreme care has had to be exercised throughout the entire mining and milling operation in order that an operating profit might be obtained. The management, however, has been able to keep costs down, with the result that at present the outlook for the property is considerably brighter than it has been in the past. This is partly due to the fact that it is now known how to mine and treat

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* The General Engineering Company (Canada) Limited, Toronto. Ont.

the ore at low costs and also that recently a somewhat better grade of ore has been developed in the mine.

The mill was designed to treat 500 tons of ore per day, but at present it is not up to capacity and is treating only approximately 400 tons of ore per day, containing gold values of approximately \$4.50 per ton at \$20.67 per ounce. Total recoveries of 95 per cent are obtained with tails of 23¢, which is exceptionally good considering the nature of the ore and the low values in the feed.

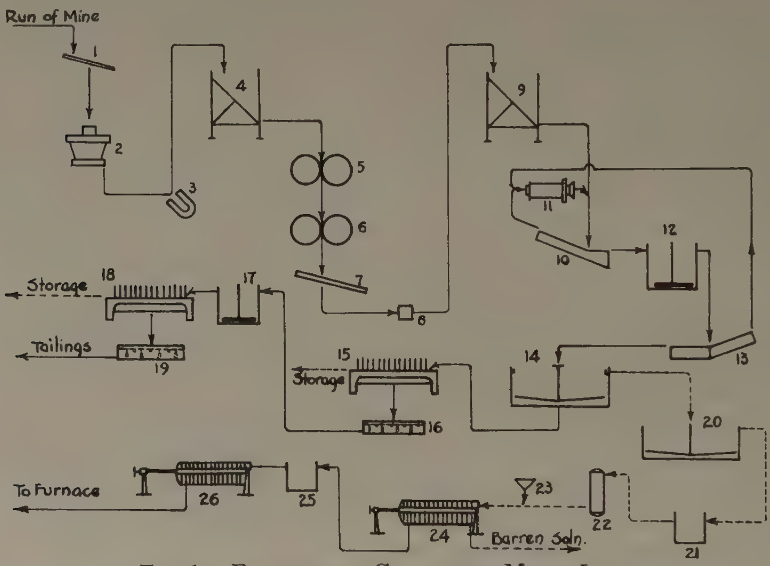

FIG. 1.—FLOW SHEET, CONIAURUM MINES LTD.

1 10-in. underground grizzly.
2 Allis-Chalmers finishing gyratory, 10 to 1½ in.
3 Cutler-Hammer magnet, 39 in.
4. Ore storage tank, 700 tons capacity.
5. 60 by 20-in. Traylor primary rolls, 1½ to ½ in.
6. 60 by 20-in. Traylor secondary rolls, ½ to −¼ in.
7. Two 4-ft. 0-in by 5-ft. 0-in Leahy vibrating screens, ¼ in.
8. Merrick model weightometer.
9. Mill storage tank, capacity 1000 tons.
10. 6-ft. 0-in by 30-ft. 0-in. Dorr duplex classifiers (2).
11. 5-ft. 0-in. by 16-ft. 0-in. Allis-Chalmers tube mills (2).
12. Four 24 by 20-ft. Dorr agitators.
13. Dorr bowl classifier, 17 ft. bowl.
14. 40 by 14-ft. Dorr tray thickener.
15. Primary American filter, 8-ft. 6-in. by 8 disks.
16. Filter-cake repulper.
17. 18 by 20-ft. Dorr agitator.
18. Final American filter, 8-ft. 6-in. by 8 disks.
19. Filter-cake repulper to tailings.
20. 32 by 10-ft. Hardinge superclarifier.
21. Two 10 by 15-ft. gold-solution storage tanks
22. 6 by 12-ft. Crowe vacuum receiver.
23. Merrill zinc-dust feeder.
24. Two Merrill precipitation presses.
25. 2 by 6-ft. precipitate acid treatment tank
26. Perrin filter press

Because of reduced tonnage treated, an excess of crushing capacity permits comparatively fine feed to the grinding mills. This in turn permits grinding to 70 per cent minus 200 mesh at comparatively low grinding costs. Ample storage bins, which are circular steel bins of 1000 tons capacity, permit the crushing plant to be operated independently of the mill, and it is thus possible to avoid peak power loads with their attendant high power costs.

The simple flow sheet (Fig. 1) has the merit, among others, of retaining the gold within the grinding circuit for prolonged treatment.

Crushing.—The mine ore is broken in a fine reduction gyratory crusher, driven by a 100-hp. motor and direct drive, from 10 to 1½ in. The product from this gyratory is conveyed by a 24-in. conveyor and a 20-in. bucket elevator to a 700-ton storage bin, from which the ore is fed to a set of 60 by 20-in. Traylor rolls, running at 1000 ft. per minute peripheral speed. The rolls are driven by two 35-hp. motors with belt drives. The product from the rolls is conveyed by a 24-in. belt conveyor to two Leahy 4 by 5-ft. vibrating screens, using ¼-in. screen cloth, and the oversize is fed to a second set of 60 by 20-in. Traylor rolls running at 1350 ft. per minute. The reduction in this step is from ½ to ¼ in. The product from these secondary rolls returns to join the feed of the vibrating screen, and a circulating load of approximately 60 tons per hour is maintained in this part of the crushing circuit. The screened product is conveyed by a 24-in. belt conveyor to a 1000-ton ore bin.

In a crushing shift of 11 hr., 470 tons of ore is crushed to minus ¼ in. from run of mine ore. Wear on manganese roll shells amounts to 0.114 lb. per ton of ore crushed and the cost of coarse crushing, screening, conveying and delivery to the mill ore bins amounts to 24.9¢ per ton of ore milled.

Grinding.—Grinding is done by a 5 by 16-ft. Allis-Chalmers tube mill, which is in closed circuit with a 6 by 30-ft. Dorr classifier. The classifier overflow at 1 to 1 dilution is pumped by a 4-in. Wilfley sand pump straight to the agitation tanks. Approximately 75 per cent of the total values in the heads is extracted in the grinding circuit.

Screen analysis on final grinding shows 70 per cent minus 200 mesh. Tube-mill ball consumption amounts to 1.7 lb. per ton of ore ground. Pocket-type liners are used and the balls are retained in the pockets, taking some of the wear, which is undetermined. Liner wear is 0.0975 lb. per ton of ore ground.

Grinding and classification costs are 17.5¢ per ton of ore milled.

Agitation and Thickening.—Agitation at 1.25 to 1 dilution for 18 hr. is done in four 24-ft. dia. by 20-ft. Dorr agitators, two units in tandem. The agitator discharge goes into a Dorr bowl classifier, which acts as a concentrator as well as a classifier, materially reducing the values in the feed to the thickeners. This concentrated product of the bowl classifier is returned to the tube mill for further grinding, as it contains approximately $3.75 in values, whereas the agitator pulp contains only approximately 60¢ (gold at $20.67 per ounce), thus showing that there is a concentration of values in the coarse product from the bowl classifier. The overflow from the bowl classifier is pumped to a 40-ft. Dorr tray thickener. Dissolution of values during agitation and thickening amounts to 16 per cent of the total values in the heads.

Filtration.—Two American eight-disk filters, 8 ft. 6 in. in diameter, are used in series with repulping between filtrations. Barren-solution wash is used on the first filter and water on the second. The final cake from the secondary filter is sent to waste. About 4.0 per cent of the original values in the heads is extracted during filtration. The filter cake from the primary filter is repulped with barren solution and is sent to a 16-ft. dia. by 20-ft. deep Dorr agitator, where an additional 4 hr. of agitation contact time is obtained at a pulp dilution of 1½ to 1. The pulp from this agitator is fed to the secondary American filter by means of a 6-in. air-lift. The tailings values show an undissolved loss of 20¢ and soluble loss of 3¢ per ton. Recoveries in various stages of the milling process are as follows:

Grinding circuit, 75 per cent extraction; agitator circuit, 16; filters, 4; total, 95.

Clarification.—The overflow from the thickener is pumped to a Hardinge sand filter for clarification. This filter, clarifying 700 tons of solution per day, gives a very clear solution preparatory to precipitation. Precipitation is done by a standard Crowe vacuum system using zinc dust. In preparing the Hardinge sand clarifier for use, the crushed rock bed is covered with sand from which all plus ½-in. material has been removed. Considerable care is taken in originally laying the sand, which is done by hand. The cost of building up the sand bottom in this clarifier amounts to approximately $225, and this must be done about once in every 18 months. The rakes of the clarifiers are revolved about three times per shift without lowering. They are lowered and a cut taken from the sand only when it is necessary to remove the fine film of lime from the surface and thus maintain the required rate of flow of solution through the sand. Screen analysis of the sand used in preparing this thickener for use is as follows: +¼ mesh, 0.0 per cent; +20 mesh. 29.0;+100 mesh, 67.0; −100 mesh, 3.5.

TABLE 1.—*Milling Costs for* 1933 *at Coniaurum Mines*
Tons of Ore Milled, 145,657

	CENTS PER TON MILLED
Crushing, screening and conveying	24.9
Tube milling and classification	17.5
Agitation and thickening	5.9
Clarification and precipitation	3.9
Reagents	8.4
Double filtration	9.3
Tailings disposal	5.4
Refining	1.2
Pumping	3.5
Total	80.0

Reagent consumption, in pounds per ton of ore milled, is: cyanide (KCN), 0.5 lb.; lime, 2.1; zinc dust, 0.0205; lead acetate, 0.0075.

Power costs at the Coniaurum property are $4.63 per horsepower per month peak load.

A summary of total milling costs for 1933 is given in Table 1. This includes heating and overhead charges.

In milling 400 tons of ore per 24 hr., as in 1933, only one tube mill and two of the four agitators are used.

Dome Mines, Limited

The Dome Mines has had a long and varied career and has survived several trying periods during its history. Discovered in 1909, the property has steadily increased in importance. It passed through a period when it was considered that the life of the mine was over; twice survived the calamity of having its surface plant completely destroyed by fire; and is now one of the most important gold producers in Canada.

Metallurgical problems have been very serious at times, but these have all been overcome, with the result that the mill is now making exceptionally fine metallurgical extractions. Blanket practice has been developed to a very high state of efficiency in the present mill and the description of this part of the flow sheet should be of considerable value to anyone familiar with gold metallurgy.

Fig. 2 shows the flow sheet of the crushing plant and mill. The run of mine ore is crushed in a Farrell jaw crusher to 4-in. size, and is then conveyed to a 5½-ft. standard Symons cone crusher, which is set for a ⅜-in. product. This product is passed over Hum-mer vibrating screens with ⅜ by ⅝-in. openings. The oversize goes to 18 by 42-in. rolls set at ¼ in. and this product returns to the Hum-mer screens. The undersize from these screens is delivered to the mill bin by a belt conveyor. Ore from the mill bin is fed by conveyors to three 8-ft. by 30-in. Hardinge ball mills, using 4-in. balls. The discharge from these goes to five Duplex D 6 by 18-ft. 4-in. Dorr classifiers, which in turn feed five 5 by 22-ft. tube mills that use No. 4 Danish pebbles. The product from the tube mills and the overflow from the classifiers is pumped to the blanket plant.

The tailings from the blanket plant are pumped to two large cones, which distribute the flow to 16 smaller cone classifiers. The spigot discharge from these cones is pumped to four Duplex model C Dorr classifiers. The rake product is fed to two tube mills, 53-in. by 22-ft. inside diameter, which use 1½-in. steel balls. The overflow from the Dorr classifiers and the product from the regrind tube mills joins the flow from the primary grinding units and is pumped to the blanket plant.

The overflow from the 16 cone classifiers flows to four 40-ft. dia. by 14-ft. 4-in. Dorr tray thickeners. The clear water overflow returns to the general mill circuit, while the underflow is pumped to four 40-ft. dia. by 14-ft. Pachuca agitators, where the pulp is agitated for about 7½ hr. This preliminary agitation increases cyanide extraction and reduces cyanide consumption. The aerated pulp then has cyanide solution added to it and is pumped to another series of Pachuca tanks, where it receives about 11½ hr. agitation in cyanide solution. The pulp then discharges into two storage or surge tanks, which in turn feed by gravity to the five Merrill filters with ninety 4-in. frames each. The unclarified solution from these presses is then pumped through Merrill clarifiers and the gold is precipitated out by standard Merrill-Crowe precipitation. The pulp, discharged from the Merrill slime press, is thickened in Dorr thickeners and pumped into the tailings-storage dam, the thickener overflow being used for sluicing out the Merrill presses.

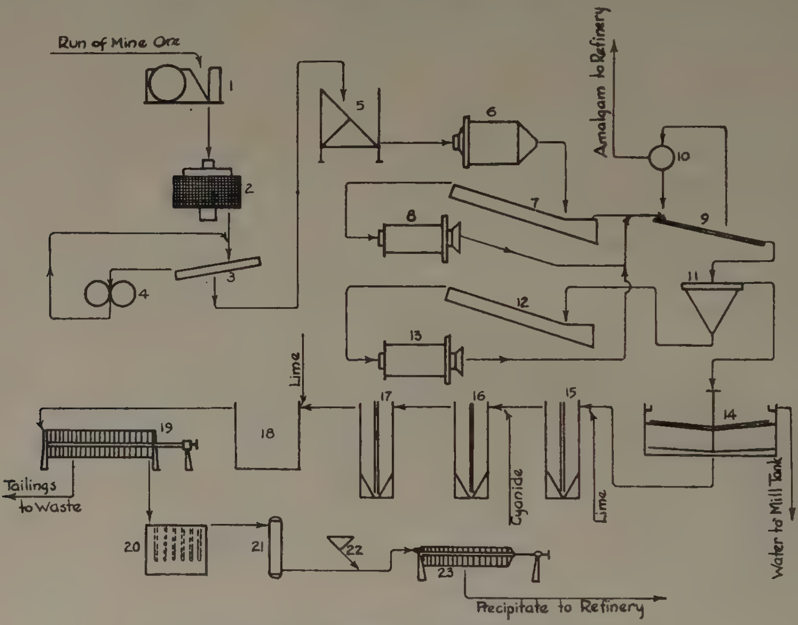

FIG. 2.—FLOW SHEET—DOME MINES LTD.

1. Farrell jaw crusher, 4-in. discharge.
2. 5½-ft. standard Symons cone crusher, ⅜-in. discharge.
3. Hum-mer vibrating screens, ⅜ by ⅝-in. openings.
4. 18 by 42-in. rolls, set at ¼ in.
5. Mill ore bin, 1800 tons.
6. Three 8-ft. by 30-in. Hardinge ball mills, 4-in. balls.
7. Five 6-ft. by 18-ft. 4-in. Dorr duplex classifiers, model D.
8. Five 5 by 22-ft. tube mills.
9. Twenty-eight 4½ by 6-ft. blanket tables.
10. Three 3 by 5-ft. amalgam barrels.
11. Two distributing cones.
    Sixteen classifying cones, ¾-in. spigot
12. Four duplex Dorr classifiers, model C
13. Two 5 by 22-ft. tube mills.
14. Four 40 by 14-ft. 4-in. Dorr tray thickeners.
15. Four 40 by 14-ft. Pachuca agitators.
16. Four 40 by 8-ft. Pachuca agitators.
17. Four 40 by 14-ft. Pachuca agitators.
18. Surge tanks, one 8 by 25 ft., one 8 by 20 ft
19. Five 4 by 6-ft. Merrill filters, ninety 4-in. frames.
20. Two 42-in. Merrill clarifiers.
21. Crowe tank.
22. Zinc feeder.
23. Three 52-in. Merrill precipitation filters

Dome Blanket Practice

In the Dome mill, destroyed by fire in 1929, free gold was removed from the circuit by a combination of amalgam plates and blankets. In the new mill an attempt was made to use an all-cyanide process but this was quickly dropped and a blanket plant installed to remove the free gold from the circuit.

The feed to the blanket tables is the unclassified product (about 60 per cent minus 200 mesh) of the primary and secondary grinding circuits. The pulp is about five parts water to one part ore. The primary load is 1500 tons, with an additional circulating load of approximately 2000 tons, making a total load over the blankets of 3500 tons daily.

There are 28 blanket tables of a combined area of 756 sq. ft., each table being 4 ft. 6 in. wide by 6 ft. long. The tables are of wood, set so that the inclination of the blankets is 1⅜ in. per foot. The corduroy blankets are 28 in. wide and cut 5 ft. long to allow for shrinkage during use. The first strip is held in place by a flat iron bar but the other two strips are held in place by lapping the upper one over the next lower one. The "Special Heavy Backed Undyed Corduroy 28-in. wide" is specially manufactured for this purpose by James Johnston, 18 London Road, Manchester, England.

The strips are placed on the table with the high sides of the cords toward the flow of the stream, thus making the necessary riffles to catch the gold. The blankets are removed and washed five times during an 8-hr. shift; three men changing, washing and looking after distribution on tables, etc. When the blankets are changed, they are folded and rolled up so as to keep the product on the inside. They are replaced by another set of blankets but before the next change are washed and rolled ready to be re-used. The washing is done in boxes, one for each shift, the blankets being merely unrolled and moved longitudinally up and down in water two or three times. This removes the greater part of the product. When blankets are discarded they are more carefully cleaned and are then treated by cyanide in a small tank until no further gold comes into solution.

The pulp is fed from launders to each table by means of a flexible rubber pipe. When blankets are to be changed, this hose can be changed to a second launder which, by means of a small air-lift, returns the pulp to the original feed stream.

Each wash tub has a pipe leading out of the bottom, so that it can be loaded directly into an amalgam barrel set directly below the tub. The amount collected in each tub in an 8-hr. shift is approximately one ton, which is the capacity of the amalgam barrels. The barrels are 31 in. in diameter by 5 ft. long and are lined with white iron liners, but barrels with cast-iron shells are preferable because they can be used without linings. Linings invariably collect and hold up amalgam. Each barrel is charged with 500 lb. of 1½-in. balls and is driven at 19 revolutions per minute.

These barrels are loaded each day about noon, lime is added and the barrels are closed and started. The real function of the grinding is to brighten all the gold so that it will amalgamate readily. At 6:30 the following morning the barrels are stopped and the requisite amount of mercury is added. The barrels are then rotated again for 1½ hr. and then dumped. The mercury and amalgam are collected, cleaned up and pressed into cakes which contain approximately 50 per cent mercury, carrying about 8 dwt. in gold per ounce. The amalgam is taken to the refinery, where it is held until sufficient accumulates for a retort.

In addition to the three men on each shift, two men on the day shift handle the dumping of the barrels and the cleaning of the amalgam.

During the first six months of 1934, the total recovery at the Dome mill was 98.6 per cent, the blanket plant giving 76.1 per cent and the cyanide plant 22.5 per cent. The tailings averaged 0.1068 dwt.

The consumption of cyanide (NaCN) in pounds per ton milled was 0.4; of lime, 2.6 lb.; zinc dust 0.044, and lead acetate 0.03.

Hollinger Consolidated Gold Mines, Limited

The Hollinger mill, the largest in the Canadian North, milled 4758 tons per day in 1933 and at present is treating 5500 tons per day. In 1933 the mill averaged $5.97 in gold values with gold at $20.67 an ounce. The ore is a siliceous schist containing about 4.2 per cent pyrite, is of a medium hardness and has a specific gravity of 2.8.

Briefly, the general plan of treatment consists of crushing, grinding in cyanide solution, classification, table concentration of the sulfides for additional grinding and more intensive cyanidation, agitation and double-stage filtration.

Table concentration recovers approximately 76.1 per cent of the pyrite in the heads to the table and gold recoveries in the concentrates are approximately 87.5 per cent of the value in the table feed. Ratio concentration of the table concentrates is about 5.95 to 1. The table concentrates, amounting approximately to 800 tons per day, contain 10.9 per cent sulfur and have a value of about $14.87 per ton. Table concentrates receive a more intensive cyanide agitation treatment than the table tails and eventually are ground to 91.4 per cent minus 200 mesh. It is interesting to note that only 0.1 per cent of the pyrite is with the plus 200-mesh material after grinding. An extraction of about 42.4 per cent of the total values in the heads is obtained during the treatment of the concentrate, of which about three-fourths is extracted in the grinding circuit. Table tails contain about 48.5¢ in gold values.

A total recovery of 96.6 per cent is being obtained with final tailings of 20.2¢ Of this final tailing 18.5 cents represents the undissolved loss and 1.7 represents the soluble loss.

Milling costs are 62.2 cents per ton which, considering the nature of the ore and the excessive grinding required on the concentrates, is a remarkably low cost and indicates the efficiency with which the mill is being operated.

Crushing

Fig. 3 shows the flow sheet of the crushing plant. The ore is crushed underground in a 48 by 60-in. Buchanan jaw crusher to 6 in. and hoisted to the ore bins on the surface. The ore is dumped from the ore skips on to a chute feeding three No. 7½ Gates gyratory crushers, breaking to 3½ in. The product from each crusher is fed to a 4-ft. dia. by 16-ft. trommel having 1-in. openings; the oversize falls to four secondary No. 5 Gates gyratory crushers breaking to 1¼ in. and is then fed to one or two 62 by 40-in. Traylor rolls breaking to ¾ in. The No. 7½ gyratory crushers are driven by a 100-hp. motor. The No. 5 gyratory crushers are driven by a 25-hp. motor. The primary rolls are driven by a 50-hp. motor on each roll. The product from each primary set of rolls runs to two 4 by 8-ft. trommels with 1-in. round openings and the oversize from the trommels is conveyed to the two secondary rolls, which are each in closed circuit with two 4 by 8-ft. trommels with 1-in. round openings.

TABLE 2.—*Crushing, Screening and Conveying Costs at Hollinger Mill*
CENTS PER TON

COARSE AND FINE CRUSHING		SCREENING AND CONVEYING, DRY-CRUSHING PLANT	
Jaw crusher	0.78	Screening	0.61
No. 7½ gyratory	1.28	Conveying	0.85
No. 5 gyratory	0.88	Dust control	0.37
All rolls	1.57	Conveying to mill ore bins	2.27
Total	4.51	Total	4.10

Total cost of crushing ore and conveying to mill bins—8.61 cents.

The undersize from the trommels is conveyed to an underground storage pocket which has a draw-off capacity of 1200 tons and thence is fed by feed rollers into weighing hoppers in 7-ton lots, the weight being automatically controlled. The hoppers are dumped into 7-ton ore cars and transported by hoist and cable up an inclined track about ¼ mile to dump over the mill ore bins.

Table 2 shows crushing, screening and conveying costs.

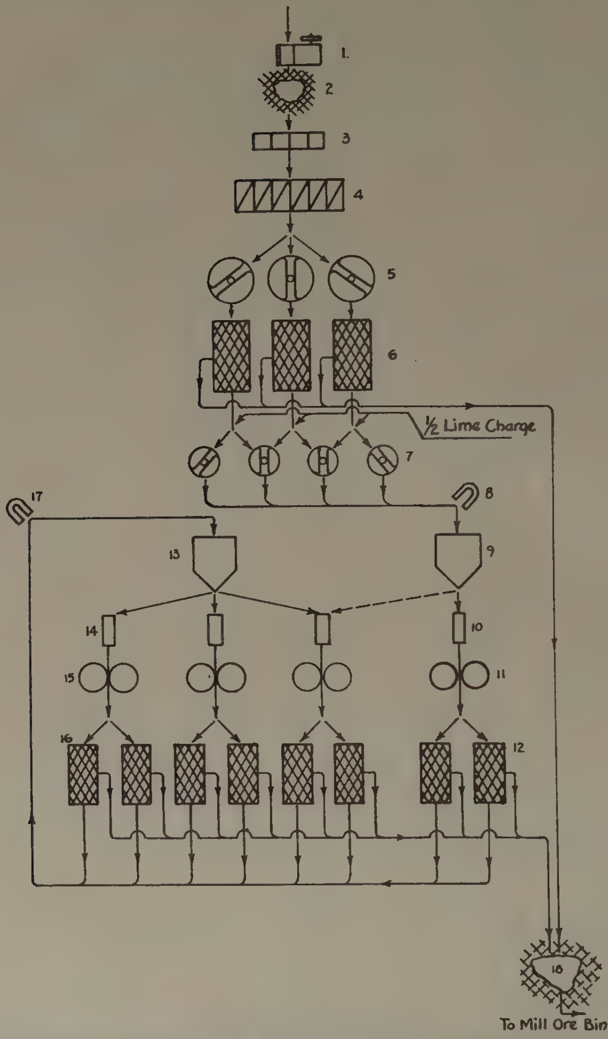

Fig. 3.—Flow sheet crushing and rolls plant, Hollinger Consolidated Gol Mines Limited.

1. Buchamnn jaw crusher, 48 by 60 in., type C.
1. Ore pocket, 1000 tons.
3. Loading station, 4 hoppers.
4. Central shaft, 4 to 7-ton skips.
5. Three No. 7½ Gates gyratory crushers.
6. Three 14 by 16-ft. trommels (Kennedy) screens
7. Four No. 5 Gates gyratory crushers
8. Two.electromagnets.
9. Feed bins to primary rolls.
10. 30-in. conveyor feed belt.
11. One 62 by 24-in. primary roll (Traylor).
12. One set of twin trommels, 4 by 8 ft.
13. Feed bins to secondary rolls.
14 Three 30-in. conveyor belts.
15 Three 62 by 24-in. secondary rolls (Taylor)
16. Three sets of twin trommels, 4 by 8 ft.
17. One electromagnet.
18. 4000-ton crushed-ore pocket

Grinding

Fig. 4 shows the flow sheet of the grinding plant and mill. The mill feed is fed from the mill ore bins by a series of five variable-speed feed rollers, the feed falling on to a 16-in. conveyor belt running along the rollers and thence to a similar belt at right angles to it, which feeds the rod mill. Grinding is done by seven 7 by 15-ft. Marcy rod mills, which are in open circuit with Dorr classifiers. The sand returned from the Dorr classifiers is in closed circuit with four 5 by 20-ft. tube mills and ten 6 by 16-ft. tube mills. The rod mills are driven by 200-hp. motors and Lennix-type belt drives. The tube mills are driven by 125-hp. motors and also Lennix drives. Grinding costs, in cents per ton of ore milled, are as follows:

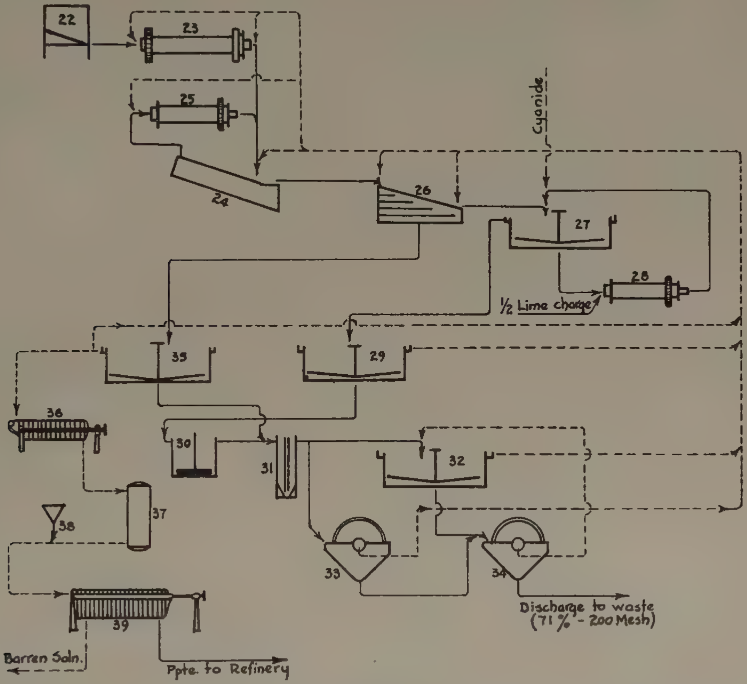

Fig. 4.—Flow sheet of cyanide plant, Hollinger Consolidated Gold Mines Limited.

22. Mill ore bins, 1200 tons draw-off capacity.
23. Seven 7 by 15-ft. Marcy rod mills.
24. Fourteen 6 by 29-ft. Dorr classifiers.
25. Four 5 by 20-ft. tube mills.
    Ten 6 by 16-ft. tube mills.
26. 102 double-deck Deister slime tables.
27. Two 12 by 6-ft. Dorr thickening classifiers.
28. Three 5 by 20-ft. tube mills.
29. Two 40-ft. Dorr tray thickeners.
30. Four 20 by 24-ft. Dorr agitators.
31. Four rows of five 15 by 45-ft. Pachuca agitators.
32. Four rows of three-step decantation (40-ft Dorr tray thickeners).
33. Six 14 by 16-ft. Oliver filters.
34. Eight 14 by 16-ft. Oliver filters.
35. Twelve 40-ft. Dorr tray thickeners.
36. Three 36-in. Merrill clarifying presses.
    One 42-in. Merrill clarifying press.
37. Vacuum receiver.
38. Zinc-dust feeder.
39. Eight precipitation presses.

Rod mill	7.32
Tube mills	6.07
Classification	1.01
Cleaning up, conveying, spillage, etc.	0.82
Total	15.22

In the grinding circuit, including pumping the primary classifier overflow to the tables, about 47.2 per cent of the values in the heads is extracted. Screen analysis of final tails shows 4 per cent plus 48 mesh and 71.9 per cent minus 200 mesh.

Ball consumption on the 5 by 20-ft. mills, using 1⅜-in. balls, is 0.432 lb. per ton of ore milled. French flints are used in the tube mills and consumption there is 1.880 lb. per ton of ore milled. Rod-mill steel consumption on 3-in. diameter rods is 0.745 lb. per ton of ore milled. Liner wear is as follows: ball mill (pocket), 0.027 lbs. per ton milled; pebble mill (Eloro), 0.086; rod mill (smooth), manganese steel, 0.165.

Agitation and Filtration

The overflow from the Dorr classifiers is pumped by 8-in. Morris pumps to double-decked Deister slime-concentrating tables, where the sulfides are taken off as a concentrate for further grinding to unlock the values, which are very finely disseminated throughout these sulfides. The table tails are pumped to primary 40-ft. diameter tray Dorr thickeners. The thickener underflow at about 54 per cent solids is pumped to Pachuca agitators, or Dorr agitators. A portion of the thickener overflow, 4737 tons, is pumped to three 36-in. and one 42-in. Merrill clarifying presses and then through a standard Crowe vacuum system using zinc-dust precipitation. The remainder of the overflow becomes part of the mill circulating solution.

The table concentrate, containing approximately 0.75 oz. in gold, is pumped to two thickening classifiers, 12-ft. diameter by 6 ft. deep, the overflow from which goes to two secondary 40-ft. Dorr tray thickeners. The comparatively clear overflow joins part of the overflow from the primary thickeners for circulating purposes. The underflow from the thickening classifiers is in closed circuit with three 5 by 20-ft. tube mills partly filled with 1⅜-in. diameter balls, thereby closing the circuit for the excessively fine grinding required on sulfide concentrates.

Some interesting cost figures in table concentration are: tabling, 2.48¢ per ton milled; regrinding concentrates, 2.51¢; conveying, pumping, agitation, etc., 0.93¢.

The underflow from the two concentrate thickeners (40 ft.) at 60 per cent solids runs to four 20-ft. dia. by 24-ft. Dorr agitators arranged in series of two's and receives 27 to 30 hr. of agitation (about 28.5 hr. average in 1933).

About one-half the lime used during the process is added at the head of the 5 by 20-ft. concentrate tube mills. All the cyanide is added ahead of concentrate-classifying thickeners. Consequently, although the main solutions in the mill titrate 0.75 lb. NaCN and 0.75 lb. protective alkalinity per ton of solution, the solution at the head of the concentrate agitation titrates 1.40 lb. NaCN and 1.30 lb. protective alkalinity.

The discharge from the concentrate agitators runs to the pump sump taking the primary thickener underflow and is agitated again with the thickened table tails.

The underflow from the primary thickeners is pumped to four rows of five 15-ft. dia. by 45-ft. Pachucas, where about 16 hr. of agitation and aeration is accomplished. The Pachuca overflow is split in two ways; part goes to primary filters and part to four rows of three-step decantation using 40-ft. Dorr tray thickeners. The discharge from the primary filters is repulped in a mechanical mixer with barren solution and joins the underflow from the decantation thickeners and this mixture is sent to the secondary filter. The filtrate from the primary filters goes directly to the mill-solution storage tank. The filtrate from the secondary filters plus some barren solution is used as diluting solution in the decantation tanks and finally reaches the mill-solution storage tank. Both water wash and barren solution washes are used on the secondary filters but only barren solution wash on the primary filters.

TABLE 3.—*Costs of Agitation and Filtration, Hollinger Mill*
CENTS PER TON MILLED

Agitation, Thickening and Pumping		Reagents		
		Consumption[a]		Costs
Thickening	0.30	Cyanide	0.489	
Agitation	0.87	Lime	2.15	7.16
Handling pulp (pumping)	1.29	Zinc dust	0.05	1.49
		Lead acetate	0.0106	

FILTRATION		PRECIPITATION AND REFINING	
Primary	1.59	Precipitation, including clarification and cleaning of presses	2.04
Secondary	2.87	Refining	1.64
Tailing disposal	1.94	Total	3.68
Pumping storage solution	0.72		
Decantation	0.80		
Total	7.92		

[a] Pounds per ton milled.

Percentage of extraction during agitation, thickening and pumping amounts to 6.8 per cent of the total recovery. Extraction during decantation and filtration amounts to 0.4 per cent.

Various costs and other data are given in Table 3.

Treatment of Gold Precipitate

The Hollinger mines refine their own bullion and have worked out an interesting flow sheet. A complete detailed description[1] of the Hollinger refining practice was published in 1931.

McINTYRE PORCUPINE MINES LIMITED

McIntyre ore consists of quartz, porphyry and schistose basalt and dacite, with a pyrite content of 3 to 15 per cent. The gold is associated with both the quartz and sulfides, predominating to a greater extent in the latter. A typical analysis of the ore fed to the mill over a period of 14 months is given in Table 9.

TABLE 4.—*Total Operating Costs, McIntyre Porcupine Mill*

	Cents per Ton		Cents per Ton
Crushing and conveying	10.70	Assaying	1.52
Flotation	36.33	Mill alterations	0.41
Cyanidation	27.34		
Refining	2.22	Total	78.52

The general scheme of treatment consists of crushing the run of mine ore in jaw and cone crushers, to 1 in., and reducing the crusher product to 3⁄16 in. by rolls in closed circuit with vibrating screens. The screen product is ground to minus 65 mesh in tube mills operating in closed circuit with single flotation cells and classifiers. Classifier overflow is floated, with elimination of a finished tailing. Concentrate from the flotation cells is reground in cyanide solution to minus 325 mesh in tube mills, operating in closed circuit with classifiers. Next comes agitation of the pulp and thickening of it, followed by three stages of filtering and washing with agitation between stages. Final residue goes to waste. Precipitation of the pregnant solution is done by the Merrill-Crowe system. The precipitate goes to the refinery and the gold bullion to the mint. Table 4 gives a distribution of the total crushing and milling costs.

Crushing

The flow sheet of the crushing plant is given in Fig. 5.

Underground Crushing.—The run of mine ore is fed to a 36 by 48-in. Traylor jaw crusher fed by a 64-in. Ross chain feeder, the crusher being driven by a 150-hp. motor and V-rope drive, and the Ross feeder being driven by a 5-hp. motor through a worm reducer, with sprocket chain gear and pinion drive. The ore drops from the crusher into a loading pocket, from which it is hoisted to the surface in 6-ton skips that dump into a 750-ton steel storage bin above the cone crusher.

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[1] M. Scott: The Treatment of Hollinger Precipitate to Produce Fine Gold. Canadian Inst. Min. and Met. (1931) **34**, 228.

Surface Crushing.—From the storage bin the ore is fed to a 7-ft. Symons cone crusher set at $\frac{7}{16}$ in., which reduces the ore to approximately 1 in. at the rate of 150 tons per hour. Manganese liners last 10 months and have a wear of 0.020 lb. per ton of ore crushed.

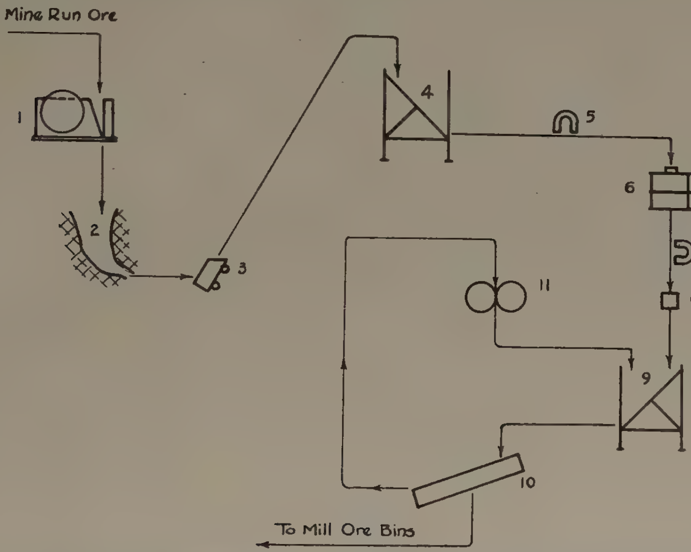

FIG. 5.—FLOW SHEET OF CRUSHING PLANT, MCINTYRE PORCUPINE MINES LIMITED.

1. Underground Traylor jaw crusher, 36 by 48 in.
2. Ore chute.
3. Skip.
4. Ore bin, 750 tons.
5. Magnet.
6 7-ft. Symons standard cone crusher.
7. Magnet.
8. Merrick weightometer.
9. Surge bin, 190 tons.
10. Six 4 by 6-ft. Hum-mer screens.
11. 78 by 18-in. Traylor Ajo-type rolls

Magnets are placed over the ore stream feeding the cone crusher and also over the conveyor handling the crusher product.

A conveyor transfers the ore from the cone crusher to the surge bin, and is in closed circuit with the rolls. It has a capacity of 400 tons per hour, handling the current ore as well as a circulating load of 190 per cent from the rolls. On this conveyor a model E Merrick weightometer is installed between the cone crusher and the point where the return product from the rolls joins the current flow, which has a capacity of 190 tons per hour and weighs all incoming ore. A Link-Belt tripper driven at 15 ft. per min. automatically discharges the ore from the conveyor to the surge bin through two chutes. The surge bin holds 190 tons and feeds the vibrating screen units through six chutes. The feed is controlled by radial gates and drum feeders.

Screening is done on six 4 by 6-ft. single-deck Hum-mer electric vibrating screens, set at 33.5° to the horizontal. Screen cloth is of special wire with 3⁄16 by 5⁄8-in. openings and has a life of approximately 70 days. Screen oversize is conveyed by a 24-in. conveyor to the rolls. This conveyor has a capacity of 400 tons per hour.

One set of Traylor (Ajo type) rolls, 78-in. diameter by 18 in., reduces all screen oversize to 3⁄16 in. The rolls are choke fed, and are set at 1⁄8 in. Each roll is driven at 125 r.p.m. by a 150-hp. motor, through V-belts, thus giving a peripheral speed of 2550 ft. per min. with new shells. Spring pressure on the roll face is 40,000 lb. per linear inch. Chrome shells 811⁄16 in. thick are used on the rolls. They have a life of approximately 95 days and wear to 2½ in. thick before slippage necessi-

TABLE 5.—*Screen Analysis of Crusher-house Products, McIntyre Porcupine Mill*[a]

Mesh	Percentages					
	Symons Feed	Symons Discharge	Screen Feed	Roll Feed	Roll Discharge	Mill Bin Feed
+6-in. ring	16.1					
+4-in. ring	7.8					
+2-in. ring	17.5					
+1½-in. ring	5.7					
+1.050 (Tyler)	3.4	1.5	0.8	1.3	0.3	
+0.742 in.	7.1	8.7	4.2	6.8	1.9	
+0.525 in.	6.4	17.2	7.3	10.4	2.9	
+0.371 in.	5.1	16.3	8.3	13.5	5.1	
Mesh						
+3	4.4	9.8	9.8	16.1	11.9	
+4	3.4	6.8	20.6	31.0	29.0	2.2
+6	3.4	7.1	16.5	16.6	20.7	14.6
+8	1.8	4.2	8.7	2.1	6.2	15.7
+10	2.0	5.2	4.9	0.5	5.6	15.2
+14	2.2	2.0	2.9	0.2	2.7	8.6
+20	1.5	2.9	2.6	0.1	2.5	8.1
+28	1.7	2.7	2.1	0.5	2.2	6.6
+35	1.4	2.7	1.8		1.7	5.5
+48	1.4	1.8	1.4		1.3	4.3
+65	1.0	1.8	1.3		0.9	3.1
+100	1.2	1.5	1.1		0.9	3.0
+150	0.7	1.0	0.8		0.7	1.9
+200	0.4	0.7	0.6		0.4	1.7
−200	4.4	6.1	4.3	0.9	3.1	9.5
	100.0	100.0	100.0	100.0	100.0	100.0

[a] Conditions: No. 7 fine Symons cone crusher; No. 390 Ton-Cap screen cloth on Hum-mer screens; rolls set to give a 3⁄16-in. product; tonnage crushed, 2100 tons in 16 hours.

tates their removal. They wear in the center without grooving. To maintain an even face, emery stones are imposed on the edges.

After passing through the rolls, the ore is returned to the surge bin feeding the screens. Finished product passing through the screens is conveyed to the mill storage bin of 4200 tons capacity, which has a live-load capacity of approximately 3000 tons. Table 5 gives a screen analysis of all products.

A crew of eleven men—six on day shift, including a foreman, and five on night shift—operate the crushing plant during two 8-hr. shifts.

A Sly dust-collecting system collects 5 tons of 82 per cent minus 325-mesh dust in two 8-hr. crushing shifts. This dust assays $2 per ton. Because of the dust-collecting system, atmosphere in the crushing plant is practically free from dust.

Crushing costs, including labor, employees' insurance, supplies, maintenance, power and general expense, are given in Table 6.

Table 6—*Crushing Costs, McIntyre Porcupine Mill*

	Wear of Crusher Parts, Lb. per Ton		Crushing Costs, Cents per Ton
Jaw plates	First set not worn out (3 years)	Symons cone crusher	2.21
		Rolls	3.86
Cone crusher	0.020	Screens	0.75
Roll shells	0.102	Conveyors	2.07
		Heating and lighting	0.36
		Supervision	1.45
		Total cost per ton	10.70

Grinding and Flotation

Fig. 6 shows the flow sheet of the flotation plant, which has a daily capacity of 2100 tons.

From the mill ore bin the ore is fed by a series of 24-in. conveyors to the grinding units. Five Allis-Chalmers 5 by 16-ft. tube mills grind the ore to minus 65 mesh. The mills revolve at the rate of 29 r.p.m. Each mill is loaded to the center with approximately 40,000 lb. of 2-in. forged steel balls and maintained at that level by adding 650 lb. of balls daily. Moisture content of the pulp varies between 30 and 35 per cent. Wave-type white iron liners, 2½ in. thick, are used, giving an approximate life of nine months.

Each mill is operated in closed circuit with a single No. 500 Denver Sub-A flotation cell and a Dorr classifier. The total pulp discharge is passed through 4-mesh screens, attached to the end of the mills; the undersize goes to the cells, and the small amount of oversize, consisting of gangue and fine steel, passes directly to the classifiers. The tailings from the cells flow by gravity to the classifiers, the concentrate going to a receiving box. From this source a 2-in. Wilfley pump, driven at

1100 r.p.m. from a lineshaft, pumps the material to the concentrate-storage tank. The cells are operated at a dilution of 1:1, made up by the addition of water to the feed and through the cones. Screen analysis of the products is given in Table 7.

The No. 500 cells have been equipped with cones operated on the hydraulic principle, making a combination machine for the purpose of removing gold that is too coarse to float, thus avoiding an accumulation in the tube-mill circuit. Every 24 hr. the tube-mill discharge is diverted to the classifiers. Water is then added for 15 min. to separate the gangue in the cells from the high-grade concentrate, after which a product consisting of sulfides and coarse gold is removed through a 4-in. plug valve equipped with a locking device. By this scheme 400 lb. of a rich material containing approximately $3,000 (gold at $20.67 per ounce) is recovered daily. This is immediately transferred to a tube mill in the cyanide circuit, with no evident increase in the value of the cyanide residue. Prior to the addition of the cones, the cells recovered 60 per cent of the total values by flotation alone. Since the introduction of the cones, extraction has increased to 75 per cent and a definite saving has been shown in the final flotation tailings.

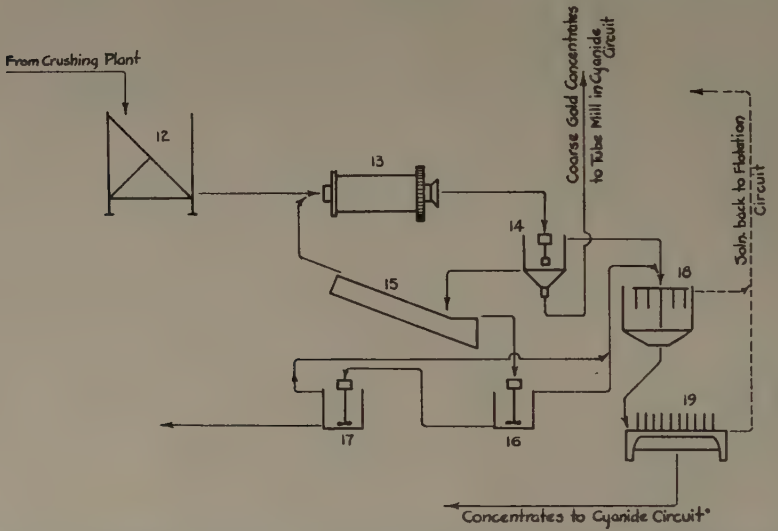
From Crushing Plant
12
13
Coarse Gold Concentrates to Tube Mill in Cyanide Circuit
Soln. back to Flotation Circuit
14
15
18
19
17
16
Concentrates to Cyanide Circuit

FIG. 6.—FLOW SHEET OF FLOTATION PLANT, McINTYRE PORCUPINE MINES LIMITED.

12. Mill ore bins, 4200 tons draw-off.
13. Five 5 by 16-ft. Allis-Chalmers tube mills.
14. Five unit flotation cells with cone bottoms.
15. Five 6 by 30-ft. Dorr duplex classifiers.
16. Eight No. 24 Denver flotation-cell units.
17. Six No. 24 Denver flotation cell units.
18. Dewatering filter surge tank with Dorr agitator mechanism.
19. Three dewatering American filters, 8 ft 6 in by 8 disks.

Five 6 by 30-ft. Dorr duplex classifiers with rakes operating at 18 strokes per minute, on a 2⅞-in. slope, return the sand by a 45° launder to the tube-mill drum feeders. The overflow at 2:1 dilution flows to a pump surge box from which it is pumped to a distributing system feeding the primary flotation cells.

Table 7.—*Screen Analyses of Tube-mill and Classifier Products, Flotation Section, McIntyre Porcupine Mill*

Mesh	Tube-mill Feed (Dry)	Tube-mill Discharge	Classifier-rake Return	Classifier Overflow
+3				
+4	2.2			
+6	14.6	0.1		
+8	15.7	0.2	0.2	
+10	15.2	0.6	0.1	
+14	8.6	0.8	0.2	
+20	8.1	1.4	0.9	
+28	6.6	3.1	5.3	
+35	5.5	7.4	12.8	
+48	4.3	10.9	24.0	
+65	3.1	12.4	31.1	6.2
+100	3.0	12.9	14.7	11.3
+150	1.9	8.6	5.1	10.7
+200	1.7	6.5	2.3	14.5
−200	9.5	35.1	3.3	57.3
	100.0	100.0	100.0	100.0

Steel consumption of balls and liners in the tube mills is as follows:

Balls	Lb per Ton	Liners	Lb. per Ton
Flotation	1.791	Flotation	0.034
Cyanidation	0.585	Cyanidation	rubber-lined

From the distributing system the pulp flows to 48 No. 24 Denver Sub-A primary cells, arranged in eight units of six cells each, one-eighth of the feed going to each unit. The impellers on the cells run at 265 r.p.m. Primary cell tails are pumped to the secondary cell distribution system and thence to 36 secondary cells. This circuit is a duplicate of the primary circuit except for the number of cells. Tailings from the secondary flotation circuit are pumped to waste through an 8-in. horizontal pipe line 1000 ft. long.

The work done by flotation is shown in the screen analysis of the primary and secondary cell tailings (Table 8). These analyses show the distribution of the values in the different sizes and their response to flotation treatment.

The chemical analysis (Table 9) of the feed and tailings in the flotation circuit indicates that the pyrite is amenable to this process, as a recovery of 98.78 per cent is shown.

Reagents.—Promoter reagents are fed to the tube-mill circuit, while frothers are added to the primary and secondary circuits and also to

TABLE 8.—*Screen Analyses and Assays of Primary and Secondary Cell Tailings, McIntyre-Porcupine*

PRIMARY CELLS

Mesh	Percentage Weight	Cumulative Per Cent	Assay	Contents, Cents
+65	6.5	6.5	$0.95	6.2
+100	12.1	18.6	0.50	6.0
+150	10.5	29.1	0.36	3.8
+200	12.3	41.4	0.40	4.9
−200	58.6	100.0	0.33	19.3
	100.0	100.0		40.2

SECONDARY CELLS

Mesh	Percentage Weight	Cumulative Per Cent	Assay	Contents, Cents
+65	6.8	6.8	$0.41	2.8
+100	12.4	19.2	0.30	3.7
+150	10.7	29.9	0.25	2.7
+200	12.2	42.1	0.29	3.5
−200	57.9	100.0	0.21	12.1
	100.0	100.0		24.8

TABLE 9.—*Chemical Analyses, McIntyre Porcupine*

	MILL FEED, AVERAGE FOR 14 MONTHS, PER CENT	FLOTATION TAILINGS, AVERAGE FOR 6 MONTHS, PER CENT
Iron pyrite	8.21	0.10
Silica	54.92	58.28
Combined water and carbon dioxide	4.32	9.10
Ferric oxide	6.28	6.79
Aluminum oxide	14.18	15.07
Calcium oxide	4.90	5.61
Magnesium oxide	4.33	2.82
Sodium and potassium oxide	2.85	2.95
Gold, per ton	$7.70	24¢
Specific gravity	2.84 dry ore	2.72 dry tailings

TABLE 10.—*Alkalinity of Flotation Solution*

	pH		pH
Lake water (Pearl Lake)	7.80	Primary-cell discharge	8.60
Tube-mill feed	8.10	Secondary-cell feed	8.80
Tube-mill discharge	8.90	Secondary-cell discharge	8.40
Classifier overflow	8.70	Filtrate from concentrate	8.30
Primary-cell feed	8.75		

the tube-mill-circuit cells. Reagent consumption is: collector, 0.18 lb. per ton; frother, 0.08 lb. per ton flotation feed.

The alkalinity of the flotation solution is shown in Table 10.

Pilot Table.—About 50 tons of tailings per 24 hr. is pumped to a 15-ft. Wilfley concentrating table. The visual operation of this table acts as a pilot to guide the flotation operator.

TABLE 11.—*Screen Analyses and Assays of Flotation Concentrates to Cyanidation*

Mesh	Percentage Weight	Assay per Ton	Contents per Ton
+65	2.5	$90.20	$ 2.26
+100	7.8	74.08	5.78
+150	10.8	70.36	7.60
+200	11.9	68.70	8.17
−200	67.0	54.57	36.56
	100.0		$60.37

TABLE 12.—*Chemical Analysis of Flotation Concentrate*

AVERAGE FOR SIX MONTHS

	PER CENT		PER CENT
Iron pyrite	64.89	Gold, dollars per ton	62.21
Silica	15.41	Silver, oz. per ton	0.01
Ferric oxide	3.17	Copper (as chalcopyrite)	0.61
Aluminum oxide	8.61	Zinc (as sphalerite)	0.71
Calcium oxide	3.19	Lead (as galena)	0.02
Magnesium oxide	2.50		

Specific gravity of dry concentrate.................... 3.80–4.20

TABLE 13.—*Flotation Costs, McIntyre Porcupine*

	CENTS PER TON MILLED		CENTS PER TON MILLED
Tube milling	12.94	Tailings disposal	2.17
Classification	1.00	Heating and lighting	0.37
Flotation machine	6.16	Supervision	1.73
Reagents	7.72		
Pumping and elevating	2.54	Total	36.33
Filtration	1.70		

Concentrate Filtration.—Concentrates from the primary and secondary cells go to the concentrate-storage tank (12 by 11 ft.) which is equipped with a Dorr agitator mechanism. The pulp is thickened in this tank to 1.7:1 dilution by twelve 6 by 3½-ft. clarifier leaves which are under a vacuum of 24 in. The clarifier leaves are cleaned daily.

The concentrates from the storage tank are pumped to three American dewatering filters, the overflow returning to the storage tank. The filters are 8 ft. 6 in. in diameter with eight disks, and are partitioned in the pulp tank, each compartment being fed from above through a 1-in. nipple from the main feed line. This prevents coarse concentrates from building up and sticking the filters.

The filter cake is washed with water to remove soluble flotation reagents. The filter bags are made of 32-oz. twill and one bag from each disk is removed for washing every 24 hr. A 24-in. vacuum is maintained on the filters. The filtrate is pumped to a steel storage tank holding 36,000 Imperial gallons.

The filter cake, at 20 per cent moisture, is repulped to 40 per cent moisture with barren solution.

The ratio of concentration by flotation is 8.98 to 1 and the concentrates have a value of $66.36.

Cyanidation

Fig. 7 shows the flow sheet of the cyanide plant, which has a capacity of 400 tons per day.

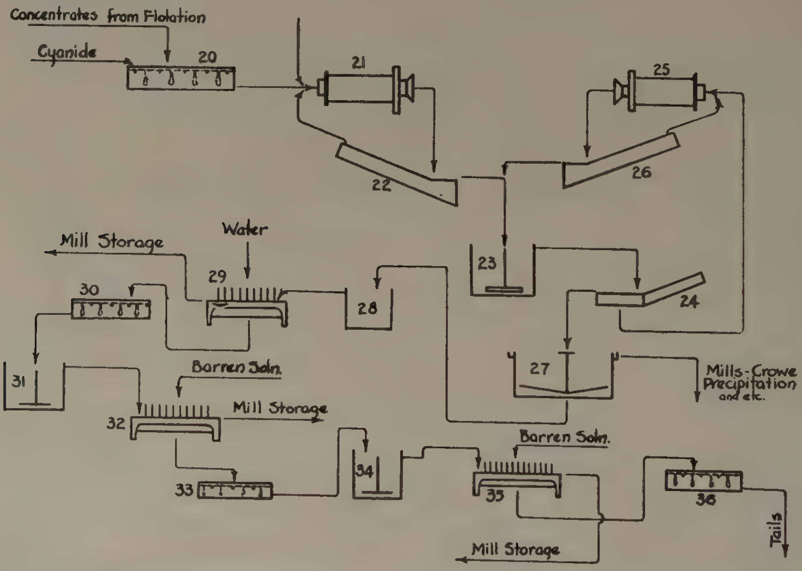

Fig. 7.—Flow sheet of cyanide plant, McIntyre Porcupine Mines Limited.

20. Three repulpers.
21. One 5 by 16-ft. Allis-Chalmers tube mill.
22. One 6 by 30-ft. Dorr classifier.
23. Four 24 by 20-ft. Dorr agitators.
24. One 20 by 6 by 30-ft. Dorr bowl classifier.
25. One 5 by 16-ft. Allis-Chalmers tube mill.
26. One 6 by 30-ft. Dorr classifier.
27. 50 by 14-ft. Dorr tray thickener.
28. Surge tank.
29. Two first-stage American filters.
30. Two repulpers.
31. Surge tank (24 by 20-ft. Dorr agitators)
32. Two second-stage American filters.
33. Two repulpers.
34. Surge tank, 24 by 20-ft. Dorr agitators.
35. One third-stage American filter.
36. One repulper.

Approximately 230 tons of concentrates per day from the flotation plant is fed to a 5 by 16-ft. Allis-Chalmers tube mill, which is in closed circuit with a 6 by 30-ft. Dorr duplex classifier. The mill is driven at

29 r.p.m. and is loaded with 30,000 lb. of 2-in. forged-steel balls, 800 lb. of new balls being added daily. Moisture content of the mill discharge is maintained at 40 per cent. Rubber liners, 1 in. thick and having a life of approximately two years, are attached to the mill shell by manganese-steel retaining bars spaced 15 in. center to center. End liners are white iron, 3 in. thick, and are replaced every 16 months.

The classifier operating at a slope of 2 11/16 in. to the foot overflows a product at 7:1 dilution. Screen analysis of the product shows 92 per cent minus 200 mesh and 80 per cent minus 325 mesh. This classifier overflow joins the overflow from the classifier of the second regrind tube mill at 1:1 dilution, thus making an average dilution of 3:1 in the agitators. The combined classifier overflow is agitated in four agitators in series. For aeration purposes a portion of the pulp is drawn from the bottom of the first agitator by a bypass arrangement and joins the current flow.

Agitation is done in four 24-ft. dia. by 20-ft. Dorr agitators, each equipped with a 36-in. discharge cone and a 6-in. air-lift, which circulates the pulp back to the tank or sends it forward to the next agitator, if so desired. Each tank is connected to the one following by 6-in. pipes located 3 ft. from the top, as well as by welded steel launders stepped down from tank to tank, thus taking care of current froth. From the final agitator the pulp goes to a Dorr bowl classifier. This classifier is 20 ft. in diameter with a reciprocating rake compartment 6 by 30 ft. Rakes move at the rate of seven strokes per minute. The bowl section, which slopes at 2 in. to the foot, has a rake speed of 1⅜ r.p.m.

As the rakes will not handle the return product under the conditions necessary to obtain the desired fineness in the overflow, the bowl is operated on the principle of a settling cone, the underflow discharging through a spigot to the feed end of the regrind tube mill, moisture content being 40 per cent. The bowl overflow is sent to the thickener at a dilution of 8 or 10:1. The overflow product is 98 per cent minus 325 mesh.

The underflow from the bowl classifier is reground in a 5 by 16-ft. tube mill in closed circuit with a 6-ft. Dorr duplex classifier. In mechanical details the mill and classifier are identical with the primary cyanide mill except that 1¼-in. balls are used, added at the rate of 800 lb. daily. Classifier overflow from this unit is kept at 1:1 dilution and joins the flow from the primary cyanide regrind mill.

The bowl overflow is split to four points on the periphery of a 50-ft. Dorr tray thickener, 14 ft. deep.

Frother is handled in the tray compartment along with a large percentage of the solids. The partially clarified solution flows down the center well to the bottom section, from which all the solution is drawn at two opposite points on the side of the thickener.

Pulp is removed at 50 per cent moisture by two 4-in. Dorr duplex diaphragm pumps. The suction of one pump is submerged in the froth on top of the thickener, while two others pump the pulp from the bottom of the tray. The remaining suction is used to take care of the pulp from the bottom of the tank. Thickened pulp flows from the diaphragm pumps to a surge tank and thence to the first-stage filters.

Three stages of filtration are used with five 8-ft. 6-in. eight-disk American filters in use, two on both the first and second stages and one on the third stage. These filters are similar to the ones used for dewatering the concentrates. Every shift two rows of bags are removed from one filter in each stage and treated with acid; this completes a cycle once in eight days.

Up until a year ago the first two stages were washed with barren solution and the final stage with water. The latest practice, however, is to wash with water on the first-stage filtering and with barren solution on the second and last stage. By this method of washing, practically no more barren solution is discarded, and, due to the water wash on the first stage, the pulp appears to be rejuvenated to a very considerable extent, with the result that the subsequent agitation and refiltration on the second and third stages dissolve an appreciable amount of values, which previously were lost in the tailings.

It is estimated that, by this change in washing, the residues have been reduced in value by 20¢ per ton. Soluble loss in the residues is about 7¢ per ton of concentrates, or less than 1¢ per ton of ore milled.

From the first stage, the cake, after being sprayed with water, is discharged at 20 per cent moisture to repulpers, where it is repulped with barren solution to 1:1 dilution. The pulp then goes to a 24 by 20-ft. surge tank, equipped with a Dorr agitator mechanism, where additional agitation time is accomplished before the second-stage filtration.

Second and third-stage filtering is done in the same way as the first except that one machine is used on the final stage. Periods occur when carbonaceous material is present in the ore and is passed to the cyanide circuit. This is indicated by the abnormal assay of froth on the agitators and thickeners, resulting in an increase in the residue value. This condition is overcome by diverting the washed residue to two six-cell Denver Sub-A flotation units, before discharging it to waste. Because of the effect of the cyanide in the prior treatment, approximately 95 per cent of the material is depressed, and the values are recovered in the form of a concentrate and returned to the cyanide circuit for further dissolution.

Pregnant solution from the thickener goes to a storage tank from which it is pumped through two 25-frame clarification presses, 42 by 42 in., using 10-oz. canvas. Canvasses are cleaned and acid-treated approximately every five days and have a life of three weeks.

Clarified solution flows to a storage tank and then to the Merrill-Crowe vacuum precipitation system. The flow of solution through the precipitation system is measured by a recording Venturi tube.

Lead acetate is added to the clarified solution previous to the addition of the zinc dust. Strong cyanide solution is also added at the same point as the zinc dust, and this aids precipitation.

Precipitation is effected at a rate of 1600 tons of solution per day in three 52-in. triangular Merrill presses, each with 22 frames. Two presses are used continuously, the third acting as a spare. Twelve-ounce canvas and unbleached sheeting are used. At intervals, the canvasses are acid-treated. They last about three months. The sheeting is burned and mixed with the precipitate at each clean-up. Every 10 days the presses are cleaned, the resultant precipitate being transferred to an acid-treating tank in the refinery. Analysis of raw precipitate is given in Table 14.

TABLE 14.—*Analysis of Raw Precipitate*

	PER CENT		PER CENT
Gold	33.29	Silica	1.48
Silver	5.76	Aluminum oxide	1.55
Copper	11.41	Calcium oxide	1.90
Lead	19.41	Magnesium oxide	0.62
Zinc	14.43	Sodium oxide	1.31
Iron	1.46		
Sulfur	6.88	Total	99.50

TABLE 15.—*Analysis of Barren Solution*

	PER CENT		PER CENT
Total solids	1.58	Sodium and potassium oxide	0.498
Total cyanogen	0.36	Magnesium oxide	0.0035
Free cyanide	0.17	Copper	0.090
Alkali (before removing CO_2)	0.19	Lead	Nil
Alkali (after removing CO_2)	0.227	Gold	Trace
Lime	0.033	Silver	Trace
Ferrocyanide	0.0054	Aluminum (Al_2O_3)	0.0036
Thiocyanate	0.210	Arsenic	Nil
Thiosulfate	0.410	Antimony	Nil
Chlorine	0.0086	Zinc	0.041

From the presses the barren solution flows to a storage tank, from which it is pumped to the filter-spray system and to supply gland solution on pumps. Surplus solution goes to the mill cyanide-storage tank. A pressure-valve arrangement on the pump discharge line maintains a pressure of 50 lb. per sq. in. on the filter sprays and glands. Analysis of the barren solution is given in Table 15.

Vacuum for filtration is maintained at 28 in., and air required for agitator air-lifts, etc. at 20 lb. pressure.

A crew of 45 men, composed of operators, mechanics, samplers, and roustabouts, is employed in the flotation and cyanide sections.

Operating costs for cyanidation, based on original tonnage, including labor, employees' insurance, supplies, maintenance, power and general expense, are given in Table 16.

Consumption of chemicals, based on a total tonnage treated from April 1 to July 31 inclusive, in pounds per ton, is: cyanide, 0.628 lb. per ton; lime, 1.106; zinc dust, 0.084; lead acetate, 0.022.

Cassel sodium cyanide is used and is added at the concentrate-regrind tube mills and agitation tanks, as well as at the zinc cone. The lime used has a solubility of approximately 85 per cent; it is slaked and added periodically to the tube-mill feed by the operators. Cyanide and lime strength in the solutions are maintained at 3.5 lb. and 0.75 lb., respectively.

All mill tailings are disposed of at 33 per cent solids, through an 8-in. wood-stave self-draining line. The line discharges at a point 1000 ft. north of the mill, where the elevation permits of gravity flow to a natural tailings pond, protected at low points by sand and gravel embankments, built to a height of 20 ft. The area of the pond is 125 acres, with a storage capacity of approximately three million tons. The solution overflow is controlled by weirs located at a central point of the lower dam, and flows to Porcupine Lake, five miles away.

TABLE 16.—*Cyanidation Costs, McIntyre Porcupine Mill*

	CENTS PER TON MILLED		CENTS PER TON MILLED
Tube milling	3.27	Precipitation	1.57
Agitation	1.05	Reagents	11.27
Classifying	0.67	Heating and lighting	0.37
Thickening	0.52	Supervision	1.74
Pumping and elevating	2.92	Mill alterations	0.41
Filtration	3.23		
Clarification	0.73	Total	27.75

Alkalinity determinations are made periodically (with a Leeds & Northrup pH indicator) on the water supply and the different solutions in the flotation circuit. The slight variations of the pH readings in the flotation circuit are due to the fact that no other compounds are used, aside from the regular collecting and frothing reagents.

Dilutions of the various products in the circuit are controlled and checked hourly by the operators. On the flotation products, with the exception of the concentrate, determinations can be made by specific gravity methods, but in the cyanide section, owing to the variation in the specific gravity of the dry concentrate, pulp-density indicators and specific-gravity methods are unreliable and dilutions must be determined by the procedure of weighing and drying the pulp.

A spacious, well lighted laboratory with the necessary apparatus is available for conducting investigations as well as checking the various steps in plant treatment.

Mill heads are sampled every half hour on the $\frac{3}{16}$-in. product feeding the tube mills. Approximately 200 lb. is taken each shift and reduced to 50 lb. by means of a Jones riffle. The sample is then sent to the assay office, where it is ground and assayed for its contents.

Flotation concentrate, flotation tailings and cyanide residue are sampled by automatic machines. Each shift, about 100 lb. of the flotation tailings and 50 lb. of concentrate and cyanide residue are taken. These samples are filtered in pressure filters, dried and sent to the assay office.

Hand samples are taken periodically by the operators from the classifier overflow and primary tailings in the flotation section, together with the classifiers, agitators, thickeners and filters in the cyanide circuit. A special sample of the cyanide residue from the third-stage filter is taken and divided into two parts. One part is dried to determine the moisture content; the other is filtered and the resultant solution assayed. From these results the soluble gold values are calculated.

Solutions in the cyanide circuit are sampled continuously by drip wires, cutting through the flow. These samples are titrated for cyanide and lime, and then assayed.

All assays are reported in dollars and cents, based on a price of $20.6718 per troy ounce for gold.

Owing to the variation of values in the mill products, different quantities are taken for assay. A charge of 3 assay-tons is used for the mill feed, whereas 10 assay-tons is required on flotation tailings. Flotation concentrate and cyanide pulp samples are roasted, ½ to 3 assay-ton charges being used.

All cyanide-solution values are determined by evaporation in lead boats, at a temperature below boiling. Duplicate assays of the pregnant and barren solutions are made, taking from 3 to 10 assay-tons.

Bullion determinations are made by standard methods. Lots of 300 mg. are weighed in triplicate; two for gold, and the other for gold and silver. Other refinery products, such as matte and slag, are assayed by the fire or wet method.

Although head samples are taken daily, they are only indicative of the production that may be expected. Actual extraction is based on bullion produced and the assay value of the automatically sampled tailings. As a general rule, the value of the bullion recovered will be 1 to 2 per cent higher than that called for by head assay.

Metallurgical results and recovery percentages are shown in Table 17.

Power contracts of the McIntyre mine are based on a cost of $50 per horsepower-year. Table 18 shows the power distribution in the mill.

A complete description of the refinery practice at the McIntyre has been published.[2]

TABLE 17.—*Metallurgical Results and Percentages of Recovery*

Metallurgical Results		Total and Stage Recovery, Per Cent	
	Value		
Heads	$7.61	*Flotation*	
Flotation tails	0.253	Unit cells	76.00
Cyanide residue	0.849	Flotation circuit	21.06
Soluble loss	0.010	Total	97.06
Combined tails	0.319	*Cyanidation*	
		Primary grinding	65.00
		Agitation and regrind	31.50
		Filters, etc.	2.20
		Total	98.70
		Total over-all recovery	95.80

TABLE 18.—*Power Distribution in McIntyre Porcupine Mill*

	HORSEPOWER		HORSEPOWER
Crusher plant (except underground crusher)	279	Refinery	17
Flotation section (includes five tube mills)	1,516	Assay office	70
Cyanide section (includes two tube mills)	558	Water supply	33
		Heating and lighting	80
		Total	2,553

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[2] J. J. Denny: McIntyre Metallurgy. *Eng. & Min. Jnl.* (1933) **134,** 472.

Acknowledgment is also made to the *Engineering and Mining Journal* for permission to quote from and use the article on the McIntyre mill; the Canadian Institute of Mining and Metallurgy for permission to quote from and use information published by the Institute; and to all others who were in any manner instrumental in helping to compile this article for publication.

Cyanidation at Kirkland Lake

By Donald F. Irvin,* Member A.I.M.E.

Amid the violent economic changes of recent years that have affected Canadian gold-mining operations so deeply, the discovery and early developments of Kirkland Lake might easily be overlooked. There was more than one "false dawn" for the district before developments finally established the fact of a superb mineralized zone extending through the present operating properties. Never far from the railway, Kirkland Lake has had the benefit of direct rail service for many years.

The pioneer discovery on which the first mill and cyanide plant was erected was the "Tough-Oakes," this property being on what is now the eastern end of the producing area; some time later, the "Lake Shore" was discovered at the present center of the district, and a small plant was built upon it also. Both of these early treatment plants were of pre-war design. Progress in gold metallurgy has caused a gradual change in cyanide practice since the early days at Kirkland Lake, which may be observed in the various plants now operating there.

From the beginning, it was recognized that Kirkland Lake ores were more responsive to cyanidation than to alternative recovery methods, and that their mineralization contained gold in very finely divided form. Although sulfides and tellurides are present, the quantity is small, being in the order of 1 or 2 per cent by weight, so that gravity concentration is undesirable, since gold is not confined to the sulfides.

Some complication is offered by the presence of tellurides, which have caused lengthy controversy over their importance in recovery methods, but which are now generally regarded as less harmful in the light of increasingly high recoveries being obtained. Nevertheless, finely divided gold intimately associated with sulfides in the presence of tellurides, and occurring in the hard Kirkland Lake porphyry ore, substantially free from natural slime-forming constituents, indicates fine grinding. This has been the universal practice although the degree of grinding has varied a little among the different mills and is considerably finer now than formerly.

The natural condition as outlined caused the establishment of fine grinding at an early stage in the development of Kirkland Lake, during the period of valuation of gold at $20.67 per ounce. With the arrival

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* Oliver United Filters Inc., New York, N. Y.

of the present gold valuation, important changes in treatment have become necessary. The gold content of tailings is now worth about 175 per cent of its former value, which gives a reason for still finer grinding to release the gold content, if that will do it, and this in turn involves new problems in all of the subsequent steps of classifying, thickening, agitating and filtering, to which may be added flotation if it forms a part of the treatment.

Factors in Choice of Treatment

It is fair to say that Kirkland Lake has never had a generally accepted uniform scheme of treatment, for there have been local differences and innovations in general metallurgical practice have been gradually adopted from time to time. Lately, there has been a division into two types: plants relying solely upon cyanidation and those that have added flotation to existing cyanide plants.

There is much division of opinion on the real value of flotation as a step in treatment at Kirkland Lake and probably a final comprehensive verdict cannot be given at this time. High recoveries (measured by final tailing assays) are being obtained by straight cyanidation as well as by combined cyanidation and flotation, so that an exhaustive study of capital cost, operating cost, net recovery, tailings loss, and expectable life of ore deposit will be needed for a true answer.

Furthermore, there are recognized differences in ore characteristics along the length of the main ore deposit of Kirkland Lake. These differences, in degree if not in kind, may justify the differences in treatment observed; also, there are observed differences in ores from the same property, although successive composite samples therefrom might retain similar characteristics.

Observation of gold content, given by production records of Kirkland Lake mines (of which all product is milled) over recent years, shows in general some diminution in grade. This is a natural concomitant, where, shaft and development facilities having been provided together with suitable treatment plants, there is an effort to mine and treat everything that will return an adequate profit per ton.

This attitude may be influenced to some degree by the selective tax recently imposed on the output of dividend-paying Canadian mines, and from which non-dividend-paying producing mines are exempt. Practically speaking, this tax falls chiefly upon the successful gold mines of northern Ontario. Just how far the tax will affect the decision as to minimum gold content in ore milled remains to be seen. Doubtless the conclusion will be based on several factors, foreign to the subject of this paper, but the result, as affecting average grade of ore milled, will be significant to the metallurgist; lower grades of ore might, or might not, yield tailings with the same gold content as the higher grades, but

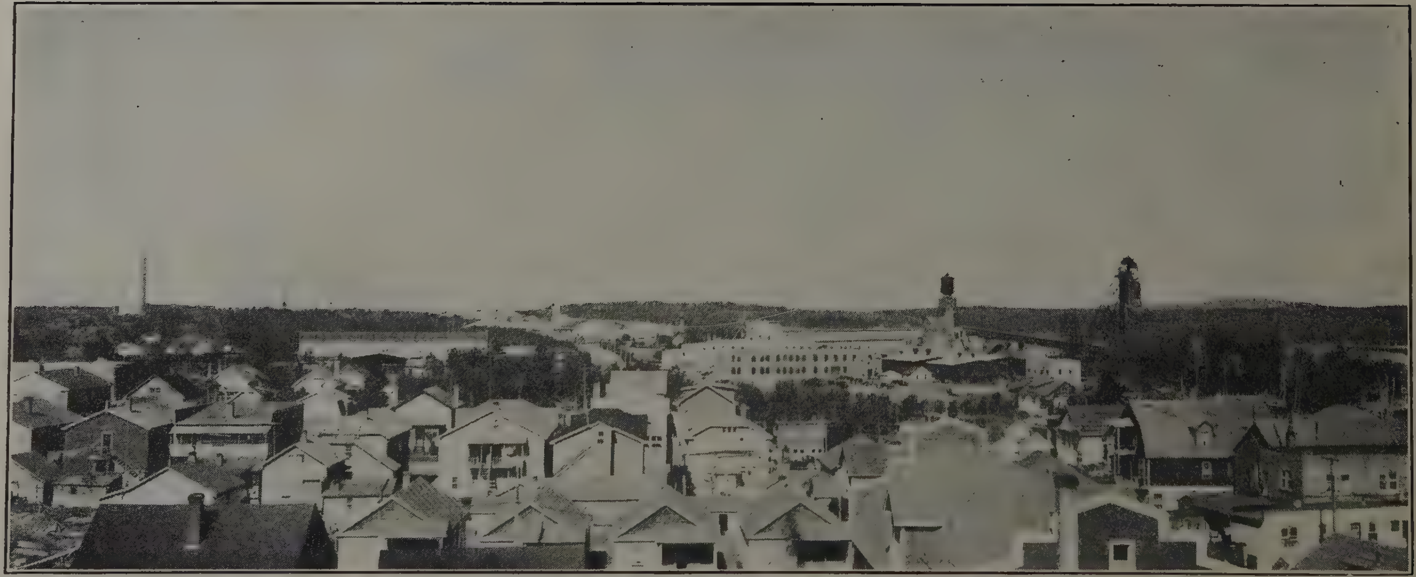

FIG. 1.—LAKE SHORE MINE AND MILL AT RIGHT, TECK-HUGHES CENTER AND KIRKLAND LAKE GOLD IN DISTANCE.

if tailings remained the same as before extraction percentages would naturally be less.

TABLE 1.—*Production Statistics for Kirkland Lake*
FIRST SIX MONTHS OF 1934 (FROM DOMINION BUREAU OF STATISTICS)

Property	Tons Milled	Gold Recovered, Oz.	Silver Recovered, Oz.	Gold Recovered per Ton Milled, Oz.
Lake Shore	415,915	241,721	51,202	0.58
Teck-Hughes	235,300	87,108	11,827	0.37
Wright-Hargreaves	179,941	113,981	24,419	0.63
Sylvanite	48,660	25,300	4,390	0.52
Macassa	33,300	16,589	1,590	0.49
Kirkland Lake Gold	30,736	8,995	1,346	0.29
Toburn	17,690	9,939	2,334	0.56

CYANIDATION ALONE AND COMBINED WITH FLOTATION

Realizing that there are some anomalous situations shown in Kirkland Lake metallurgical practice, one may group the plants in two classes: (1) straight cyanidation and (2) cyanidation with flotation. In the former are found Toburn, Sylvanite and Teck-Hughes; in the latter, Wright-Hargreaves, Lake Shore, Kirkland Lake Gold and Macassa.

Toburn uses, with some modifications and modern additions, the pioneer plant of the old Tough-Oakes built in pre-war days. Sylvanite and Teck-Hughes are of relatively late construction, their present plants dating from 1926 and 1930 respectively.

The flotation group, naturally, is very recent. Wright-Hargreaves has a completely rebuilt installation, which began work in 1933 and Macassa had no treatment plant at all before last year. On the other hand, Lake Shore built the second small plant at Kirkland Lake but its present plant —the largest by far at Kirkland Lake—represents steady and gradual growth in tonnage and no single date can in fairness be assigned to it, though its flotation annex has been in use about two years.

Kirkland Lake Gold began with a small cyanide plant of decantation type, which has been gradually equipped with later adjuncts found in newer plants. It is now installing a small flotation annex, which will be notable in that the concentrate will be shipped to smelter without treatment. The decision to ship flotation concentrate without treatment is a step toward simplicity, if net results obtained thereby are satisfying; freight and treatment schedules on concentrate are said to be more favorable than in the past, and direct shipment avoids any further complication of treatment methods in the cyanide plants.*

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* Since this paper was written, flotation at Kirkland Lake Gold has been suspended after a period of trial, but further metallurgical studies are expected by C. W. Dowsett, advisory metallurgical engineer.

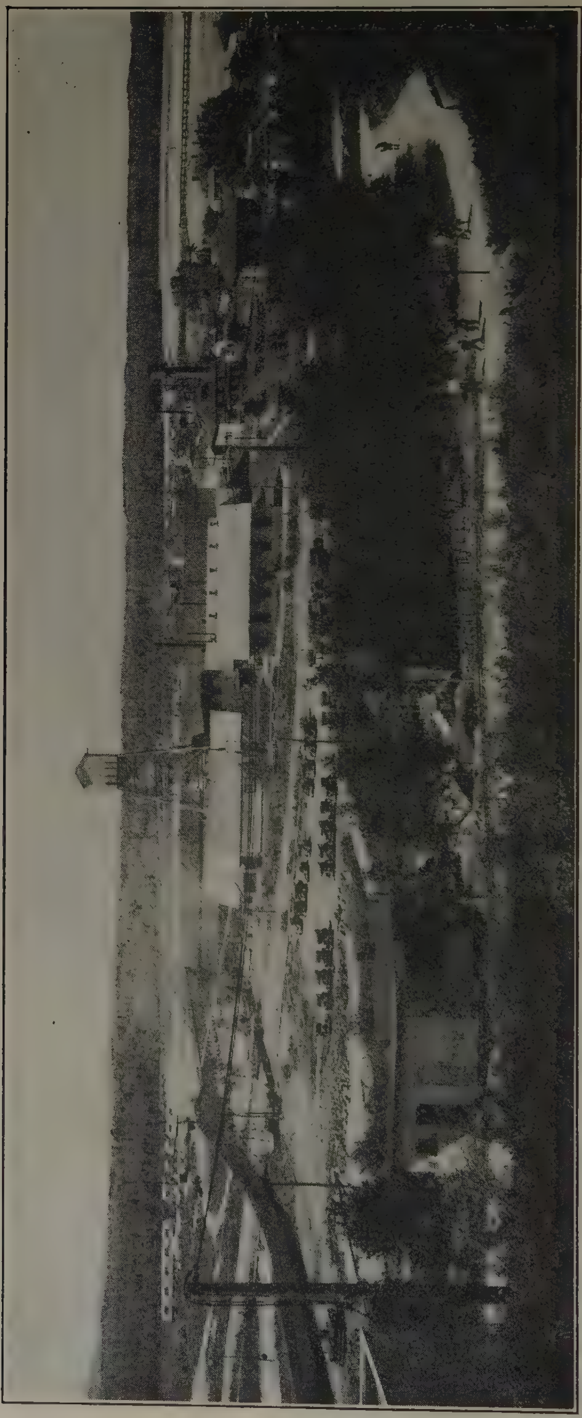

FIG. 2.—TECK-HUGHES MINE AND MILL.

The flotation plants now operating at Macassa, Lake Shore and Wright-Hargreaves have tried separate treatments of concentrate and rejecting as tailings the residues from such treatment. They have also tried returning these residues to the main flow of pulp for re-treatment. Besides this variation, they have tried flotation prior to cyanidation of main pulp, as well as floating after main pulp has been cyanided. These sequences have a bearing on the progressive accumulation of certain objectionable constituents of the pulp (when concentrates are returned after treatment) vs. over-all tailings losses.

It is doubtful whether one solution of the problem can be applied to all plants. Each plant probably requires some special variation.

An interesting change in common practice has been tried at Macassa, where alkalinity of cyanide solution is in part gained by substitution of ammonia for lime. Some lime is used and the combined alkalis are said to have given especially good results. However, this step is still being studied.

Any consideration of flotation practice at Kirkland Lake should note the very small amount of sulfides present—from 1 to 2 per cent. This causes a meager original froth, with considerable nonsulfide material. On the whole, the flotation equipment is relatively small. Various well-known types of flotation cells have been and are being used, such as Fagergren, Denver and Kraut, and a number of flotation reagents also. The choice of the latter seems endless; at one flotation plant in Quebec Lifebuoy soap is used, for the sake of one of its soluble constituents.

If concentrates are returned to the main pulp after separate cyaniding, this is tacit admission of incomplete extraction on the first passage. In one instance, it is claimed that the sulfides unattacked by cyanide again float, but that those of which the surfaces are pitted where gold particles have been dissolved do not float and pass out with the tailing.

Time consumed in passing through the plant varies with the straight cyanide plants and the combined types. The former can obtain excellent recoveries when a sufficient period of contact exists, yet the combined type of plant reports high recovery in much shorter time.

It is fair to say that flotation and cyanidation of the concentrate is not yet established firmly as a step economically desirable at all Kirkland Lake plants. A great deal of research has been done and still is in progress on this problem at all the plants and by another year one may expect with some confidence to learn definite conclusions on this question.

Crushing, Grinding and Treatment of Pulp

The mills of Kirkland Lake follow generally well established methods as to crushing, grinding and regrinding, differing chiefly in the type of grinding units, which are either ball or rod mills with secondary tube-mill units. Gyratory crushers and tube mills are of standardized type while the secondary crushing uses Symons cones, and regrinding is done by Marcy, Hardinge and Allis-Chalmers ball or rod mills. Especially interesting grinding media are the forged steel balls made in Canada from discarded steel rails. Cast balls also are used.

Classification of pulp is done by Dorr classifiers, of varied type and size, the largest modern Dorr classifiers being in the recently installed grinding units at Wright-Hargreaves and Sylvanite.

Methods of pulp treatment employ the usual steps of agitation, thickening and filtering. Agitation is done by various methods; many Dorr agitators of conventional design are in use; in several of the later mills, turboagitators and Wallace agitators have been installed. The two latter rely upon induction of atmospheric air into the pulp, through vortices caused by rotation of the impeller or by turbulence caused by rotor and stator rings.

Thickening is done in most plants by Dorr thickeners, which in some cases are of the multiple-tray type. Suction thickening by Genter thickeners using filter elements has been in use for about 1½ years at Wright-Hargreaves.

Cyanide Practice

The earlier mills employed a series of Dorr thickeners to effect cyanide treatments by countercurrent decantation. Later, the use of filters to complete recovery was found necessary, and this gradually brought about the present practice, which is the use of two-stage filtration with two stages of thickeners generally preceding the two-stage filtration.

Cyanide slime filtration is performed in all Kirkland Lake plants by Oliver filters except at the Teck-Hughes mill, where American disk filters are used in the larger section and Olivers in the smaller section, and at Lake Shore, where Americans are used on primary filtration and Olivers on secondary filtration.

Some plants repulp the filter cake discharged from the primary stage, sending this pulp directly to the second stage, while others prefer to introduce some agitation in dilute pulp and follow with thickening before sending to second-stage filtering.

Oxidation of the pulp by atmospheric air drawn through the filter cake is recognized at Kirkland Lake as a valuable aid to extraction of gold, and operation of filters is always arranged to make the most of this effect.

The very clean character of Kirkland Lake ores gives high capacity to cyanide filters, although this rate is considerably exceeded by the rate obtained on less finely ground ore in plants of the Porcupine district.

At all the mills the filter tailings are repulped and discharged by centrifugal slime pumps through wood-stave pipe lines to tailing ponds. This is necessary because of the severe winter climate and the low relief of the topography at Kirkland Lake.

Precipitation is by zinc dust, using Merrill-Crowe equipment of well-known type, and clarification of the pregnant solutions may be by Merrill presses, or by vacuum suction leaves.

Finer grinding, which now may be 85 per cent minus 325 mesh, has caused somewhat more difficulty in handling the pulp than when pulp screened 90 to 95 per cent minus 200 mesh.

The melting and refining of gold precipitate follow well established practice elsewhere. Some matte of high grade is formed, and occurrence therein of copper, tellurium and selenium is reported.

Obviously, in plants of the type described, there is much pumping of slime pulp; for this work the Wilfley centrifugal sand pump is generally used, although trials in local mills of soft rubber-lined centrifugal slime pumps have been started.

Plants at Kirkland Lake

Outwardly the Kirkland Lake plants are unusual in appearance, owing to the flat topography and to the cold climate, which causes the use of asbestos sheeting or shingles as an outer covering. The low temperatures in winter, and the increased extraction gained by heating cyanide solution to 80° or 90° F., produce an unpleasant humidity in these plants in cold weather, while in summer the low pitch of the roofs makes the plants too warm for comfort in spite of ventilation.

Advantage is taken of the heat produced by grinding and by the motors, as at Teck-Hughes, to supply the heat required to warm the building in winter.

Water and power supply are both adequate; the former from adjacent lakes, and the latter from the Hydro Electric power-system network of northern Ontario, which has a substation at Kirkland Lake. The Canada Northern Power Corporation reports gross sales of 299,066,656 kw-hr. for first seven months of 1934, and gross income of $2,343,358 for the same period—which gives an average figure for power costs.

During the 20 years of cyanide plant operation at Kirkland Lake there has accumulated a large tonnage of tailings. With the exception of the small Toburn tailing dump, and the new Macassa plant, nearly the entire remainder from all the plants lies in one compact mass, filling the bed of what was once Kirkland Lake—formerly a large body of water adjoining the town.

The tailings of earlier years are of a grade suited to re-treatment, and this step was projected seriously two or three years ago. Extensive plans were made, involving cooperative effort of two or more of the Kirkland Lake companies, but in spite of profits indicated, the project was not carried out.

The size of the present company plants measures the opinion of their respective boards of directors as to the economic outlook, and into it enter many factors, such as continuity and grade of ore, discovery of parallel or new ore chutes, what may be found in the so-called "south break," or shear zone; while the establishment or maintenance of present gold prices has enormous importance. Gradual increase of nonoperative charges, such as taxation of all kinds and silicosis compensation, also have serious importance. Fortunately, ore has been proved at great depth by several Kirkland Lake mines, so that anxiety over bottoming the deposit is not yet a matter for much concern.

In no other region save South Africa or West Australia is there found such a closely built group of numerous modern cyanide plants treating such quantities of ore—over 5000 tons daily—and the opportunities for observation and study of cyanidation are unique.

FIG. 3.—WRIGHT-HARGREAVES MINE AND MILL.

Toburn, Wright-Hargreaves and Teck-Hughes might be selected as typifying three different treatment methods. Toburn represents the decantation type of cyanide plant to which filtration recently has been added as a final step, treating an excellent grade of ore and making very good extraction. Teck-Hughes is the largest plant at Kirkland Lake operating on a straight cyanidation basis. It also obtains an excellent extraction, operating with notable smoothness. Wright-Hargreaves typifies the newest cyanidation plus flotation method and is the largest plant at Kirkland Lake committed to this method solely. Certain unique results have been obtained in it, and though the grade of ore treated is excellent, the tailings have become consistently low in value, with a very high percentage of extraction.

Kirkland Lake is indebted to Wright-Hargreaves for an active research campaign which has cleared up several points in question in the local metallurgical problem. Lake Shore also has under way a program of research, which is expected to solve certain questions on general treatment applicable to Kirkland Lake conditions, so that a statement regarding treatment methods at this great plant may be expected somewhat later. For that reason, no details of the very interesting present Lake Shore practice can be included here.

TECK-HUGHES MILL

The Teck-Hughes mill operates in two sections; the new portion of three units, completed in 1931, which handles 1000 tons daily, and the older portion, built before 1926, which handles 270 tons daily.

The ore is crushed before being conveyed to the storage bins in the mill building proper. There are two crushing units, at the Central and the South shafts, as well as an underground crusher station. In these units, a series of jaw crushers, bar grizzlies, vibrating screens and Symons cone crushers reduce crude ore to about ¾ in. size. This final size of product was chosen as best suited to the rod mills, which receive it for final grinding in the mill.

Each unit of Teck-Hughes mill has two regrinding units, each containing a 4 by 10-ft. Marcy rod mill and a 5 by 16-ft. tube mill. The rod mill is in closed circuit with a 3 by 21-ft. 8-in. Dorr simplex classifier, although a part of the sands produced go directly to the tube mill. The classifier overflow joins the tube-mill discharge and enters an 18-ft. Dorr bowl classifier.

Although the grinding section at Teck-Hughes does not conform in each item to what might be held conventional apparatus at other Kirkland Lake mills, the excellent results obtained show that the methods used have justified themselves economically.

As might be expected, the grinding circuit accounts for a high extraction of gold content (about 70 per cent), the large bulk of the gold being found in the finest product. It is this characteristic of Kirkland Lake ore that has driven the plant operators to ever finer grinding, to liberate and recover the undissolved gold in the tailings, which cannot be allowed to escape at present gold valuation.

From the bowl classifiers, a finished product is sent at 4 to 1 dilution, to be agitated in a 32 by 18-ft. Dorr agitator. This 5½-hr. treatment period before thickening includes abundant air introduced into the pulp at two points in all Teck-Hughes agitators besides the central air-lift column. The final result is considerably increased dissolution of gold, which otherwise would not, and did not, occur in primary thickener overflows, until prior pulp agitation was provided.

After agitation, the pulp flows to a pair of 26 by 14-ft. Dorr tray thickeners, which deliver pregnant solution to the clarifiers and underflow of pulp at 59 per cent moisture to secondary agitation.

Here it should be noted that agitation is continuous during 16 or 17 hr. in a series of four 24 by 16-ft. Dorr agitators, and extraction continues to the end of the agitation period, because pulp is diluted to 70 per cent moisture, with barren solution, when beginning agitation. It was found that extraction halted in a few hours, with pulp at 55 to 60 per cent moisture.

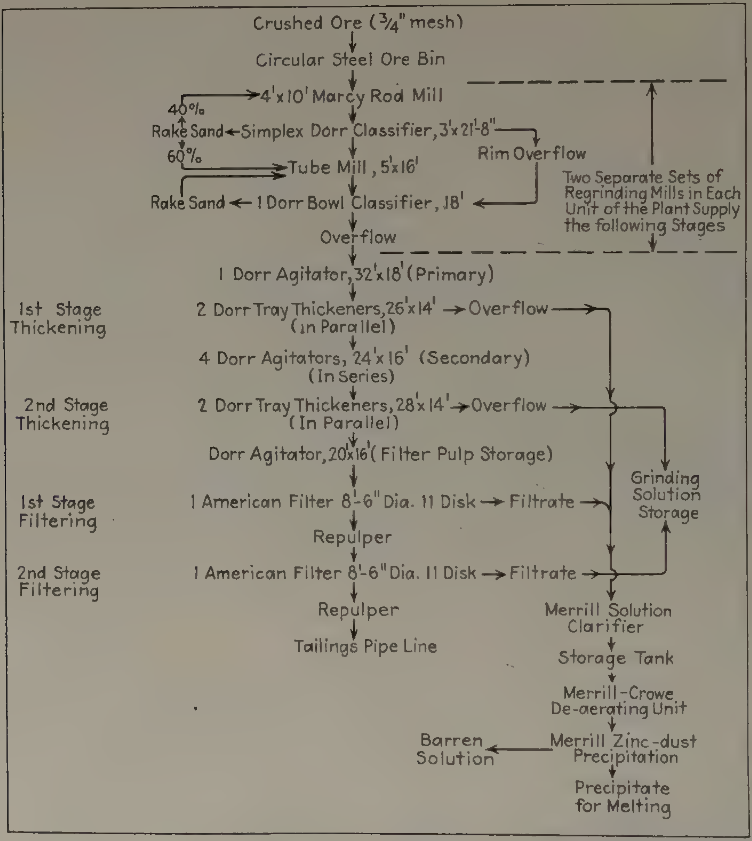
Crushed Ore (3/4" mesh)
Circular Steel Ore Bin
4'x10' Marcy Rod Mill
40%
Rake Sand
Simplex Dorr Classifier, 3'x21'-8"
60%
Tube Mill, 5'x16'
Rim Overflow
Rake Sand
1 Dorr Bowl Classifier, 18'
Two Separate Sets of Regrinding Mills in Each Unit of the Plant Supply the following Stages
Overflow
1 Dorr Agitator, 32'x18' (Primary)
1st Stage Thickening
2 Dorr Tray Thickeners, 26'x14' (in Parallel)
Overflow
4 Dorr Agitators, 24'x16' (Secondary) (In Series)
2nd Stage Thickening
2 Dorr Tray Thickeners, 28'x14' (In Parallel)
Overflow
Dorr Agitator, 20'x16' (Filter Pulp Storage)
Grinding Solution Storage
1st Stage Filtering
1 American Filter 8'-6" Dia. 11 Disk
Filtrate
Repulper
2nd Stage Filtering
1 American Filter 8'-6" Dia. 11 Disk
Filtrate
Repulper
Tailings Pipe Line
Merrill Solution Clarifier
Storage Tank
Merrill-Crowe De-aerating Unit
Barren Solution
Merrill Zinc-dust Precipitation
Precipitate for Melting

FIG. 4.—TECK-HUGHES GENERALIZED FLOW SHEET FOR ONE UNIT TREATING 300 TO 350 TONS DAILY.

The dilute pulp after agitation is sent to two 28 by 14-ft. Dorr tray thickeners, which turn off a circulating mill solution and a thick pulp to filter storage in a 20 by 16-ft. Dorr agitator.

Ample capacity is an admirable feature of Teck-Hughes design, while the frequent and sustained agitation periods provide the necessary contact and aeration for dissolution.

Filtering and washing of the cyanided pulp is done by two 8-ft. 6-in. 11-disk American filters, arranged in series with repulping of cake from

No. 1 filter to feed No. 2 filter. The repulped cake from No. 2 filter is pumped to tailing storage through a wood-stave pipe line.

The Teck-Hughes plant is notable for cleanly and "smart" operation, the working floor being high above ground, near top of tanks, leaving the ground floor to the pump installations. Supervision is aided by the plant design, and while the subdivision of the mill and its grinding department gives rather small units, excellent flexibility in operation results as a definite benefit.

WRIGHT-HARGREAVES MILL

Two facts combine to make the present plant practice at Wright-Hargreaves highly interesting: one, that it is the largest newly designed rebuilt plant at Kirkland Lake, and the other that it includes several examples of the more recent developments in metallurgical practice.

Some of the steps are still under study to determine the exact optimum under the conditions set in the Wright-Hargreaves mill, among them being pulp densities, circulating load and lime content of solution in the grinding circuit. The final pulp discharged from the grinding circuit is now 99 per cent through 200 mesh and 82 to 84 per cent through 325 mesh—an excellent example of the extreme fineness now required in these cyanide plants of late, which may be compared with the former 80 to 90 per cent through 200 mesh that was regarded as typical fine grinding not so long ago.

A magnetic pulley installation in the tube-mill circuit has eliminated a considerable amount of fine tramp iron, believed to be harmful as a reducing agent in the cyanide solution, as well as a space-filling burden in the grinding mills, and in the bottoms of the flotation cells.

Reduction in power consumed is reported for the rubber-lined slime pump; a definite statement on its performance soon will be of interest.

In preparing the pulp for flotation, the addition of copper sulfate destroys the inhibitory effect of cyanide on flotation, and at the same time it precipitates any last trace of dissolved gold, making it possible to recover it in the following flotation.

Sampling of mill pumps and solutions is done by an automatic Telechron-controlled system. This affords a good check on precipitate yield.

The complete transformation and enlargement of the former mill at Wright-Hargreaves from a rambling wooden structure to the present steel-framed and asbestos-shingle-covered building, without hindrance to production, is a testimonial to careful planning and skilful execution.

Although the accompanying flow sheet (Fig. 5) might seem at first somewhat complicated, it should be noted that it accomplishes treatment of the nonsul pofidertion of the ore in notably short time, and the small tonnage of the concentrate, containing the bulk of value in the ore, is given separately a long intensive treatment.

As to extraction and costs, it may be said confidently that they are highly creditable. A complete account of Wright-Hargreaves operation, prepared by its staff, is expected soon, in which full tabular data may be expected.

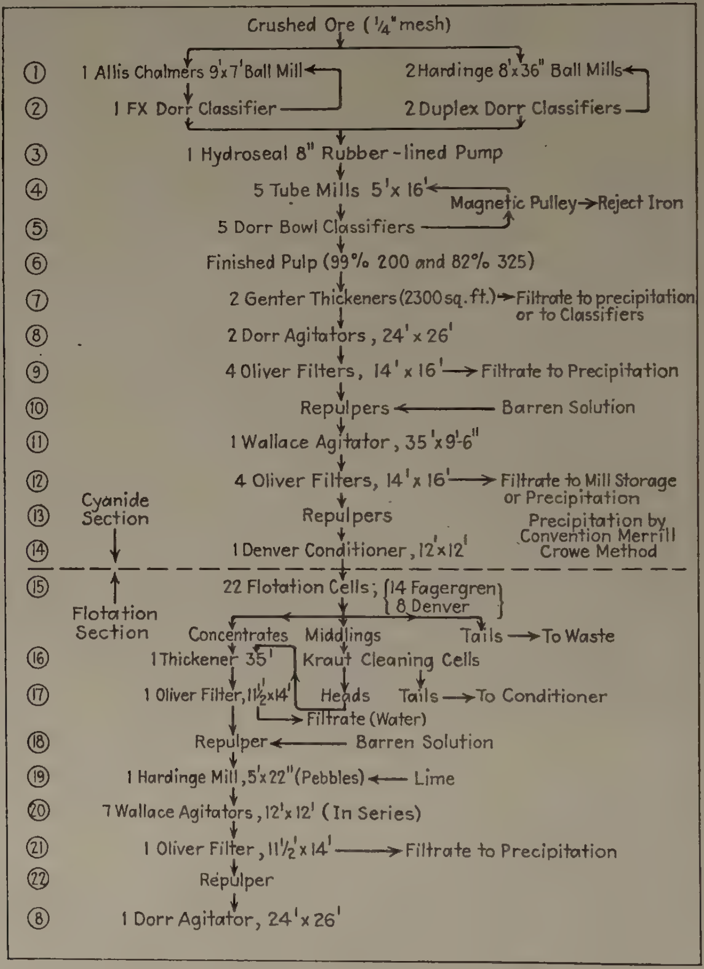
Crushed Ore (¼" mesh)
① 1 Allis Chalmers 9'x7' Ball Mill
2 Hardinge 8'x36" Ball Mills
② 1 FX Dorr Classifier
2 Duplex Dorr Classifiers
③ 1 Hydroseal 8" Rubber-lined Pump
④ 5 Tube Mills 5'x 16'
Magnetic Pulley→Reject Iron
⑤ 5 Dorr Bowl Classifiers
⑥ Finished Pulp (99% 200 and 82% 325)
⑦ 2 Genter Thickeners (2300 sq. ft.)→Filtrate to precipitation or to Classifiers
⑧ 2 Dorr Agitators, 24' x 26'
⑨ 4 Oliver Filters, 14' x 16'→Filtrate to Precipitation
⑩ Repulpers ← Barren Solution
⑪ 1 Wallace Agitator, 35'x9'-6"
⑫ 4 Oliver Filters, 14' x 16'→Filtrate to Mill Storage or Precipitation
⑬ Repulpers
Cyanide Section
Precipitation by Convention Merrill Crowe Method
⑭ 1 Denver Conditioner, 12'x12'
⑮ 22 Flotation Cells; {14 Fagergren 8 Denver}
Flotation Section
Concentrates
Middlings
Tails→To Waste
⑯ 1 Thickener 35'
Kraut Cleaning Cells
⑰ 1 Oliver Filter, 11½'x14'
Heads
Tails→To Conditioner
Filtrate (Water)
⑱ Repulper ← Barren Solution
⑲ 1 Hardinge Mill, 5'x22" (Pebbles) ← Lime
⑳ 7 Wallace Agitators, 12'x 12' (In Series)
㉑ 1 Oliver Filter, 11½'x 14'→Filtrate to Precipitation
㉒ Repulper
⑧ 1 Dorr Agitator, 24'x 26'

FIG. 5.—WRIGHT-HARGREAVES GENERALIZED FLOW SHEET.

VARYING CONDITIONS

Significant plant data and assay values of Kirkland Lake cyanide products have been omitted intentionally, because there has been a general shifting of objectives by all plant managements during the past year. Consider the chief cause—revaluation of gold. This compels finer grinding to avoid "higher" losses in tailings, and has disturbed the existing mechanics of treatment throughout. At the same time, ore reserves must be reappraised for the same reason, and "ore" treated may differ physically somewhat from normal. The new tax on gold from dividend-paying mines has imposed a third set of influences upon production and treatment.

Still unsettled questions exist as to the wisdom of adding flotation in all cyanide plants at Kirkland Lake, and as to the way in which it may be applied to best advantage. Also, the incidence of taxes (all others than bullion tax mentioned) and silicosis-compensation charges are worthy of serious study.

Therefore, comparison of extraction percentages, washed-tailing assays (whether in ounces or "dollars") and costs is risky. The 0.50-oz. ore that gives 98 per cent extraction, and the 0.30-oz. ore that gives 93.4 per cent may represent equal metallurgical skill and effort.

ACKNOWLEDGMENTS

The best characterization of cyanide metallurgical work at Kirkland Lake is that it is being carried on by an energetic, competent group of engineers who are aware of the varied conditions mentioned and who are as a group obtaining excellent results on an ore requiring specialized treatment and with high average content of finely disseminated gold.

Thanks are due to these same engineers for their courtesy in providing data on their problems of plant operation:

Plant	Manager	Mill Superintendent	Metallurgical Consultant
Kirkland Lake Gold		John Dixon	C. W. Dowsett
Lake Shore	A. L. Blomfield (Managing Director)	Hayden Rood	
Macassa	G. A. Howes	J. M. Carter	H. W. Shoemaker
Sylvanite	C. E. Rodgers	G. H. Cliff	
Teck-Hughes	D. L. H. Forbes	H. N. Read	
Toburn	M. W. Hotchkin	C. A. Randall	
Wright-Hargreaves	M. W. Summerhayes	Malcolm Black	H. Vincent Wallace

Dissolution of Gold and Silver in Cyanide Solutions

By George Barsky,* S. J. Swainson,† Member A.I.M.E., and Norman Hedley†

The cyanidation of precious-metal ores is a complex chemical process. Numerous reactions occur, some of which cause an undesired consumption of alkali and of cyanide. A knowledge of these reactions, their products, and the factors that influence the rates at which they proceed, should be of value in working out the most economical conditions for cyanidation; but at present, much of this knowledge is lacking.

This paper is confined to a study of some of the fundamentals of cyanidation. At the start of such an investigation it is necessary to simplify the problem under consideration by eliminating as many variables as possible. The results are then, of course, not immediately applicable to practice, but it is hoped in further work to round out the information and thereby make it of more practical value. For convenience in presentation, the paper has been divided into two parts, one devoted to a study of the reaction between cyanide and gold or silver, the other to a consideration of the factors that determine the rate of dissolution.

I. REACTIONS INVOLVED IN DISSOLUTION OF GOLD AND SILVER IN DILUTE CYANIDE SOLUTIONS

Ever since the cyanide process for gold and silver extraction was introduced there has been discussion of the equation representing the reactions involved. In undertaking a detailed study of these factors, it was felt that the most logical first step was to determine, if possible, what reactions could theoretically take place when gold and silver are dissolved by dilute cyanide solutions.

It is possible to determine whether or not a given reaction will theoretically take place by calculating the free energy of the reaction. This free-energy value for any given reaction is the algebraic sum of the free energies of formation of each of the reactants and products, and, if these values are known, the calculation is simple. (A chemical change always involves an energy change and the amount of energy change involved is the factor determining whether a given reaction will take place or how far it will go. In chemistry we consider what is known as the "free energy"; that is, the maximum amount of energy that can be utilized as

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work, or be transformed into a form of energy other than heat. This energy can be measured by means such as we have used here.)

When an attempt was made to calculate the free energy of the various proposed equations for dissolution of gold and silver in cyanide, it was found that the free energy of formation of the complex gold-cyanide ion is not given in the literature. The free energies of formation of the complex gold-cyanide and silver-cyanide ions were determined therefore by a method that is briefly described here. These data have made it possible to calculate the free-energy changes in the various reactions proposed and to point out which of these reactions are theoretically possible under ordinary cyanidation conditions.

In their simplest form the reactions for the dissolution of metallic silver and gold in dilute cyanide solutions are:

$$Ag + 2CN^- = Ag(CN)_2^- + (-) \qquad [1]$$
$$Au + 2CN^- = Au(CN)_2^- + (-) \qquad [2]$$

Elsner,(1)* MacLaurin,(2) Christy,(3) and Bodlaender(4) have shown that in the cyanide process these reactions will not proceed without absorption of oxygen or some other accompanying reduction reaction. In the presence of oxygen the dissolution of gold by cyanide may be expressed by the following equation:

$$4Au_{(S)} + 8CN^- + O_{2(aq)} + 2H_2O = 4Au(CN)_2^- + 4OH^- \qquad [3]$$

This is known as Elsner's equation. The reaction for silver is analogous.

In the presence of a compound such as cyanogen bromide, gold and silver will dissolve in cyanide without oxygen, in which case the reactions may be expressed as follows:

$$2Au + 4CN^- = 2Au(CN)_2^- + 2(-) \qquad [4]$$
$$BrCN + 2(-) = Br^- + CN^- \qquad [5]$$

Combining equations 4 and 5 gives:

$$2Au + 3CN^- + BrCN = 2Au(CN)_2^- + Br^-$$

Janin(5) has suggested that gold is dissolved with liberation of hydrogen according to the following equation:

$$2Au + 4KCN + 2H_2O = 2KAu(CN)_2 + 2KOH + H_2$$

or, in the ionic form,

$$2Au + 4CN^- + 2H_2O = 2Au(CN)_2^- + 2OH^- + H_2$$

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* References are at the end of the paper.

Bodlaender(4) has suggested the following:

$$2Au + 4KCN + 2H_2O + O_2 = 2KAu(CN)_2 + 2KOH + H_2O_2$$

the hydrogen peroxide formed being used in the reaction

$$2Au + 4KCN + H_2O_2 = 2KAu(CN)_2 + 2KOH$$

The over-all equation is thus the same as Elsner's. Expressed in terms of ions these reactions are

$$2Au + 4CN^- + 2H_2O + O_2 = 2Au(CN)_2^- + 2OH^- + H_2O_2$$

and

$$2Au + 4CN^- + H_2O_2 = 2Au(CN)_2^- + 2OH^-$$

As pointed out above, whether the reactions proposed are possible can be calculated from the free-energy changes involved. The free energies of formation of all reactants and products are known except that of the aurocyanide ion.

Randall and Halford(6) studied the solubility of silver chloride in aqueous hydrogen cyanide. From the solubility data they calculated the free energy of formation of $Ag(CN)_2^-$, $\Delta F_{298\cdot1} = 72{,}047$ cal. They state, however, that "the acid concentration produced in these equilibria is so small that an accurate determination of the equilibrium constant is difficult."

As the literature contains no data regarding the free energy of formation of $Au(CN)_2^-$, it was necessary to determine this value. At the same time it was considered advisable to check Randall and Halford's value for the free energy of formation of argentocyanide ion by another method.

METHOD

The method used involved a measurement of the electromotive force of the chains

$$Au_{(S)}, \begin{matrix}(Au(CN)_2^-)\\(CN^- \quad\quad);\end{matrix} \text{Sat. KCl, } HgCl_{(S)}, Hg$$

$$Ag_{(S)}, \begin{matrix}(Ag(CN)_2^-)\\(CN^- \quad\quad);\end{matrix} \text{Sat. KCl, } HgCl_{(S)}, Hg$$

Annealed gold and silver wire was used. Oxygen was excluded from the gold and silver electrodes by passing nitrogen through the cyanide solutions. The chain reactions are:

At the silver-argentocyanide pole:

$$Ag_{(S)} + 2CN^- = Ag(CN)_2^- + (-)$$

At the gold-aurocyanide pole:

$$Au_{(S)} + 2CN^- = Au(CN)_2^- + (-)$$

At the calomel pole:

$$HgCl + (-) = Hg_{(L)} + Cl^-$$

The electromotive forces of the silver-argentocyanide and gold-aurocyanide poles are thus determined, since the e.m.f. of the saturated calomel electrode against normal hydrogen electrode is known. Then the dissolution potentials at unit activity are:

$$E_0 = E_{(Ag)} + \frac{RT}{F} \ln \frac{A_{Ag(CN)_2^-}}{A^2_{CN^-}} \qquad [6]$$

$$E_0 = E_{(Au)} + \frac{RT}{F} \ln \frac{A_{Au(CN)_2^-}}{A^2_{CN^-}} \qquad [7]$$

when $E_{(Ag)}$ and $E_{(Au)}$ are the electromotive forces of the silver and gold poles.

In highly dilute solutions the activities may be assumed to be equal to the molalities, in which case

$$E_0 = E_{(Ag)} + \frac{RT}{F} \ln \frac{M_{Ag(CN)_2^-}}{M^2_{CN^-}} \qquad [8]$$

$$E_0 = E_{(Au)} + \frac{RT}{F} \ln \frac{M_{Au(CN)_2^-}}{M^2_{CN^-}} \qquad [9]$$

EXPERIMENTAL

Distilled water was used in preparing all solutions. The NaCN-NaAu(CN)$_2$ solutions were prepared by dissolving pure gold foil in a sodium cyanide solution. The NaCN-NaAg(CN)$_2$ solutions were prepared by dissolving purified AgCN in a solution of sodium cyanide. The gold and silver content of the solutions was determined by the Chiddy fire-assay method; the free cyanide by the Liebig titration using potassium iodide as indicator.

The nitrogen was passed through alkaline pyrogallol solution, water, and finally a cyanide solution of the same concentration as that in which the electrode was immersed, before it entered the pole (Fig. 1). The temperature was maintained at 25° C. ± 0.05°.

The time-voltage curves (Fig. 2) for two silver-argentocyanide chains studied show that equilibrium was quickly established after which the e.m.f. was constant. These e.m.f. include the junction potential difference between saturated KCl and 0.005 molar NaCN. We believe that this liquid junction potential difference will probably not exceed 5 millivolts, which would not affect the final value for the $\Delta F°$ of $Au(CN)_2^-$ and $Ag(CN)_2^-$ by more than one per cent.

Molality $Ag(CN)_2$	Molality CN^-	Chain e.m.f.
(1) 0.00985	0.00440	0.5060
(2) 0.00493	0.00408	0.5180

Taking the value of 0.2458 as the value for the e.m.f. of the saturated calomel electrode against the normal hydrogen electrode(7) at 25° C., the e.m.f. of the silver-argentocyanide pole are 0.2602 and 0.2722 volt, respectively for 1 and 2, then by equation 8:

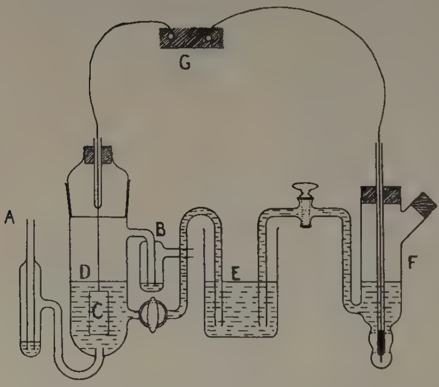

Fig. 1.

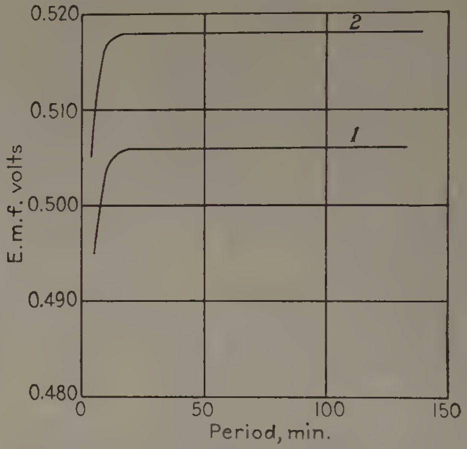

Fig. 2.

Fig. 1.—Apparatus for measuring electromotive force in gold-cyanide reactions.

A. Nitrogen inlet.
B. Nitrogen outlet.
C. Gold electrode.
D. $NaAu(CN)_2$ solution.
E. Saturated KCl solution.
F. Calomel electrode.
G. Potentiometer.

Fig. 2.—Time-voltage curves for two silver-argentocyanide chains.

$$(1)\quad E_0 = 0.2602 + 0.05915 \log \frac{0.00985}{(0.0044)^2}$$
$$= 0.2602 + 0.1601$$
$$= 0.4203 \text{ volt}$$
$$(2)\quad E_0 = 0.2722 + 0.05915 \log \frac{0.00493}{(0.00408)^2}$$
$$= 0.2722 + 0.1462$$
$$= 0.4184 \text{ volt}$$

The difference between these two values of E_0 is 0.0019 volt, hence we are justified in using the average value, which is 0.4194 volt.

Then for the reaction:

$$Ag + 2CN^- = Ag(CN)_2^- + (-)$$
$$\Delta F^\circ_{298.1} = -EF = -(0.4194 \times 23{,}074)$$
$$= -9677 \text{ calories}$$

From the value for the free energy of CN^- (39,370) the free energy of formation of argentocyanide ion may be calculated as follows:

$$Ag_{(S)} + 2C_{(graph)} + N_{2(g)} + (-) = Ag(CN)_2^-$$
$$\Delta F^\circ_{298.1} = 69{,}063 \text{ calories}$$

This value is 2984 calories lower than the value of 72,047 calories reported by Randall and Halford.(9)

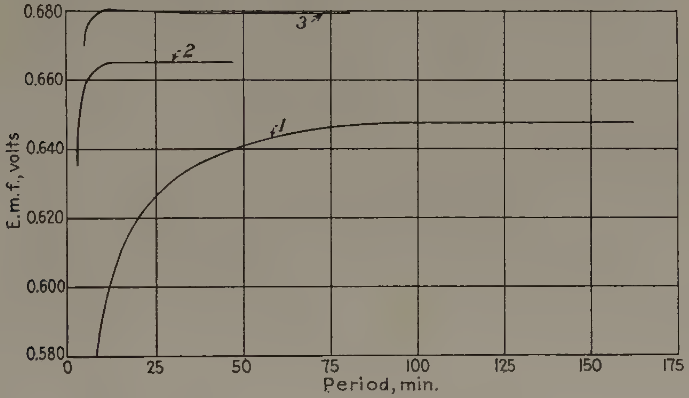

FIG. 3.—TIME-VOLTAGE CURVES FOR THREE GOLD-AUROCYANIDE CHAINS.

Fig. 3 shows the time-voltage curves for three gold-aurocyanide chains studied. The data may be summarized as follows:

Molality $Au(CN)_2^-$	Molality CN^-	Chain E.M.F.
(1) 0.00462	0.00510	0.6475
(2) 0.00245	0.00506	0.6650
(3) 0.00183	0.00510	0.6790

from which the e.m.f. of the gold-aurocyanide poles are 0.4017, 0.4192, and 0.4332, respectively, for 1, 2 and 3.

Then by equation 9:

$$(1)\ E_0 = 0.4017 + 0.05915 \log \frac{0.00462}{(0.00510)^2}$$
$$= 0.4017 + 0.1331$$
$$= 0.5348 \text{ volt}$$

$$(2)\ E_0 = 0.4192 + 0.05915 \log \frac{0.00245}{(0.00506)^2}$$
$$= 0.4192 + 0.1172$$
$$= 0.5364 \text{ volt}$$

$$(3)\ E_0 = 0.4332 + 0.05915 \log \frac{0.00183}{(0.00510)^2}$$
$$= 0.4332 + 0.1093$$
$$= 0.5425 \text{ volt}$$

Since c and d check very closely, we will take the average value for $E_0 = 0.5356$ volt. Then for the reaction:

$$\begin{aligned} Au + 2CN^- &= Au(CN)_2^- + (-) \\ \Delta F^\circ_{298.1} &= -EF = -(0.5356 \times 23{,}074) \\ &= -12{,}358 \text{ calories} \end{aligned}$$

From which the free energy of formation of aurocyanide ion is

$$\begin{aligned} Au_{(S)} + 2C_{(graph)} + N_{2(g)} + (-) &= Au(CN)_2^- \\ \Delta F^\circ_{298.1} &= 66{,}382 \text{ calories} \end{aligned}$$

With these data on the free energies of formation of $Au(CN)_2^-$ and $Ag(CN)_2^-$, the equilibrium constants of the various equations proposed have been calculated.

For Elsner's equation:

$$4Au + 8CN^- + O_2 + 2H_2O = 4Au(CN)_2^- + 4OH^-;\ K = 10^{66.0}$$

similarly:

$$4Ag + 8CN^- + O_2 + 2H_2O = 4Ag(CN)_2^- + 4OH^-;\ K = 10^{58.1}$$

These equilibrium constants show that in the presence of oxygen the reactions will proceed practically to completion; in other words, until practically all the cyanide has been consumed, or all of the metal dissolved.

In the same way the equilibrium constant for Janin's equation has been calculated:

$$\begin{aligned} 2Au + 4CN^- + 2H_2O &= 2Au(CN)_2^- + 2OH^- + H_2;\ K = 10^{-9.9} \\ 2Ag + 4CN^- + 2H_2O &= 2Ag(CN)_2^- + 2OH^- + H_2;\ K = 10^{-13.9} \end{aligned}$$

The equilibrium constants are so very unfavorable that the formation of hydrogen may be considered impossible under ordinary cyanidation conditions. For Bodlaender's first equation:

$$\begin{aligned} 2Au + 4CN^- + 2H_2O + O_2 &= 2Au(CN)_2^- + H_2O_2 + 2OH^-;\ K = 10^{16.0} \\ 2Ag + 4CN^- + 2H_2O + O_2 &= 2Ag(CN)_2^- + H_2O_2 + 2OH^-;\ K = 10^{12.1} \end{aligned}$$

Similarly, for Bodlaender's second equation:

$$\begin{aligned} 2Au + 4CN^- + H_2O_2 &= 2Au(CN)_2^- + 2OH^-;\ K = 10^{49.8} \\ 2Ag + 4CN^- + H_2O_2 &= 2Ag(CN)_2^- + 2OH^-;\ K = 10^{46.0} \end{aligned}$$

Here again the equilibrium constants are favorable, therefore the reactions proposed by Bodlaender are possible.

It is interesting to note, in this connection, that Bodlaender actually found that hydrogen peroxide was formed, and he was able to account for approximately 70 per cent of the theoretical amount of hydrogen peroxide that should be formed according to his equation.

In view of this it would appear that Bodlaender's equations express the true reactions that take place when gold and silver are dissolved in dilute cyanide solutions. Although Elsner's equation gives the same final products as the sum of Bodlaender's equations, it is not strictly correct, because it does not express the intermediate step involving the production of hydrogen peroxide, the formation of which Bodlaender proved.

II. RATE OF DISSOLUTION OF GOLD OR SILVER IN CYANIDE SOLUTIONS

Effect of Cyanide Concentration on Rate of Dissolution of Gold

According to MacLaurin,(12) the rate of dissolution of gold in cyanide solutions attains a maximum in passing from concentrated to dilute solutions. His work shows that this maximum is reached at a solution concentration of 0.25 per cent KCN, or an equivalent of 0.19 per cent NaCN.

Julian and Smart(13) made a number of exact determinations of the solubility of gold in dilute solutions containing 0.01 to 0.10 per cent KCN. They found that the rate of dissolution of gold (or, the units by weight dissolved in equal intervals of time) increased rapidly with increase in strength of solution up to and including 0.10 per cent KCN. They state, however, that "if the time factor is left out of the question, as where gold dissolves rapidly, but can be washed out of the ore only slowly, the most efficient strength of solution is from 0.07 to 0.09 per cent KCN." This corresponds to a solution containing 0.05 to 0.07 per cent NaCN.

In the early days of cyanidation it was customary to use solutions containing as much as 0.25 per cent NaCN for gold ores. This was particularly true in plants that ground relatively coarsely and employed sand leaching. With the arrival of fine grinding and all-slime practice, the strength of solution was reduced and today most cyanide plants treating gold ores use solutions containing 0.05 per cent NaCN or even less. Stronger solutions do not seem to hasten the dissolution of gold or improve the extraction. As the chemical and mechanical loss of cyanide is much higher with strong solutions, it is obviously desirable to maintain the solution at the minimum strength consistent with good extraction. As previously stated this strength in practice appears to be approximately 0.05 per cent NaCN, or one pound of NaCN per ton of solution.

Because the majority of cyanide plants treating gold ores use cyanide solutions considerably below 0.25 per cent, an investigation was undertaken of the rates of dissolution of pure gold in solutions containing from zero to 0.50 per cent NaCN.

Apparatus

An apparatus was designed to permit accurate determinations of gold loss without removing the gold from the cyanide solution. This was accomplished by suspending the gold in the cyanide solution by means of a fine glass thread, the upper end of which was attached to the stirrup hook of an analytical balance (Fig. 4). The cyanide-solution container consisted of a glass cylinder 2 in. in diameter and 4½ in. high, with a ground-glass cover. In the center of the cover was a hole 0.12 in. (3 mm.) in diameter through which passed the glass suspension thread. The cover was also provided with an air-inlet tube dipping below the surface of the solution and curved upward at the end to cause the air bubbles to sweep the surface of the gold. The temperature was maintained constant by means of a water jacket through which water from a bath was circulated

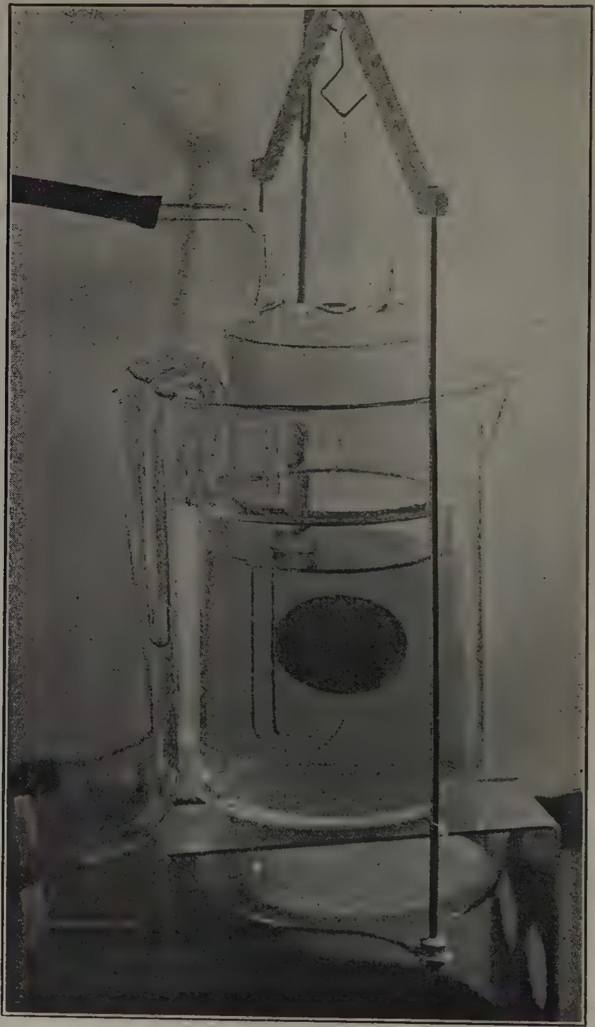

Fig. 4.—Apparatus for determining gold loss without removing gold from solution.

by pump and siphon. The complete arrangement was supported upon a bridge standing over the balance pan.

The air supply was first passed through a scrubbing tower containing sodium cyanide of the same strength as that in the dissolution cylinder. This scrubbing tower was immersed in the water bath. From the scrubbing tower the air passed through a flowmeter by means of which the air supply was maintained constant.

Pure gold foil, 0.001 in. (0.025 mm.) thick, in the form of disks of 1.5 sq. in. (10 sq. cm.) in area, was used.

Procedure

The gold disks were annealed at a temperature of 650° C. for 1 hr. after which they were treated for 30 min. with a 0.05 per cent sodium cyanide solution through which a stream of air was passing. After washing with water, alcohol and ether, the disks were transferred to a stoppered bottle. This preliminary treatment with sodium cyanide was carried out to have a fresh gold surface in contact with the solution at the beginning of the test, thereby eliminating any effect of passivity. (Subsequent tests have shown this preliminary treatment to be unnecessary. Treated and untreated gold showed the same rates of dissolution.)

The scrubbing tower was filled to the required height with sodium cyanide solution, and 100 c. c. of the solution under investigation was transferred to the apparatus and aerated for 30 min. A weighed disk of gold was then attached to the glass thread, the cover of the apparatus removed and the thread inserted through the center hole. The cover was replaced, the gold now being in the cyanide solution, and the apparatus, together with the water jacket, transferred to the balance case. The other end of the glass thread was attached to the stirrup hook, the water connection put in place and the air line attached. After the apparatus was arranged so that the glass thread did not foul the sides of the aperture in the cover, the gold, suspended in the cyanide solution, was weighed. The air was turned on and adjusted so that the flowmeter scale read 10 divisions. (This flow of air gave sufficient agitation so that the rate of diffusion exceeded the maximum rate of dissolution.) A time reading was taken immediately. The water pump was turned on and water at a temperature of 25° C. circulated through the water jacket. At the end of 15 min. the air supply was cut off, the water pump stopped, and another weighing made.

This same procedure was carried out every 15 min. for 1 hr., and every ½ hr. thereafter. At the end of the test the gold was removed from the apparatus, washed with water, alcohol and ether, then weighed on the assay balance. The difference between this weight and the original weight gave the total gold loss, which was used as a check against the cumulative loss.

The bubbling of the air through the cyanide solution caused a certain amount of spray, some of which was deposited on the glass thread. To obtain an accurate measurement of the gold dissolved, this deposition was determined and a correction applied to the weighings. A correction was also made for the difference between the weights of gold in cyanide solution and in air.

No alkali was added. The pH of all solutions was over 9.0.

Experimental Results

Using the procedure outlined, tests were run with solutions of various concentrations, from 0.01 to 0.50 per cent NaCN. Several check tests were run on each strength of solution. The results recorded are the averages of these tests. From the data obtained, curves were plotted to show the relationship between cumulative loss and time (Fig. 5). Some of the data are given in Table 1. The maximum rate of dissolution was obtained with 0.05 per cent solution of NaCN.

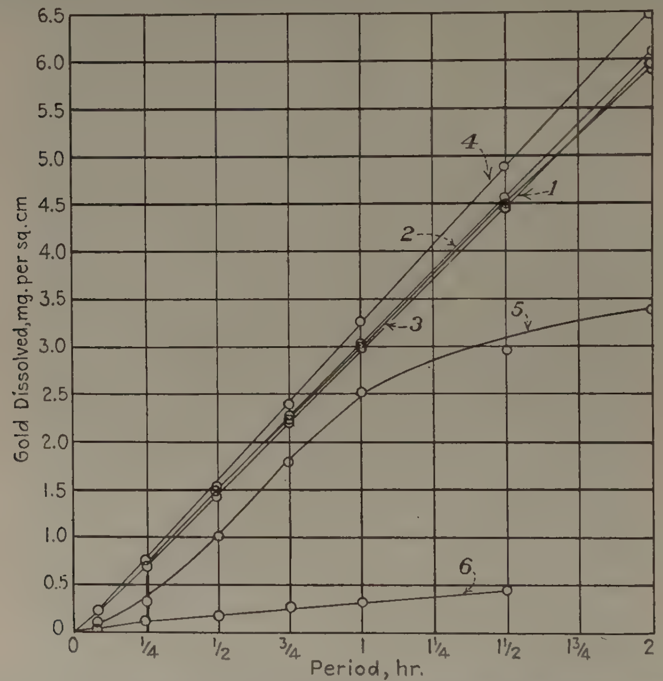

FIG. 5.—CUMULATIVE GOLD LOSS IN VARIOUS STRENGTHS OF CYANIDE.

MacLaurin has stated⁽¹²⁾ that the amount of gold dissolved in potassium cyanide is proportional to the absorption coefficient of oxygen in such solutions. To determine whether there is any relationship between cyanide strength and oxygen dissolved, a series of tests was run in which the oxygen was determined in air-saturated cyanide solutions varying

TABLE 1.—*Relative Amounts of Gold Dissolved during First Hour*

NaCN in Solution, Per Cent	Gold Dissolved in 1 Hr., Mg. per Sq. Cm.	Refer to Fig. 5, Curve No
0.500	2.943	1
0.250	3.007	2
0.100	2.986	3
0.050	3.251	4
0.025	2.513	5
0.010	0.338	6

from 0.01 to 2.00 per cent NaCN. The method used for the determination of oxygen in the cyanide solutions was that outlined by Weinig and Bowen.(14) The results are recorded in Table 2. The solubility of oxygen is practically unaffected by the concentration of cyanide. The fact that strong cyanide solutions do not dissolve gold more rapidly than weak solutions is not due to lower oxygen solubility in such solutions.

TABLE 2.—*Amounts of Oxygen in Cyanide Solutions*

NaCN, Per Cent	Oxygen per Liter, Mg.
0.0 (distilled water)	8.20
0.01	8.25
0.025	7.90
0.05	8.02
0.10	7.96
0.25	8.29
0.50	8.33
2.00	8.36

In these experiments, the maximum rate of dissolution of pure gold was reached at 0.05 per cent NaCN, which corresponds with concentrations used in modern mill practice.

EFFECT OF CYANIDE CONCENTRATION ON RATE OF DISSOLUTION OF SILVER

To determine the effect of strength of sodium cyanide solutions upon the rate of dissolution of silver a series of tests was run with cyanide solutions varying from 0.50 to 0.01 per cent NaCN. The silver used was in the form of foil, 0.001 in. (0.025 mm.) thick. The apparatus and procedure were the same as described in the preceding section. The results obtained are shown by the curves in Fig. 6, and some of the data are given in Table 3.

TABLE 3.—*Effect of Cyanide Concentration on Dissolution of Silver*

NaCN in Solution, Per Cent	Silver Dissolved in 1 Hr., Mg. per Sq. Cm.	Refer to Fig. 6 Curve No.
0.486	1.290	1
0.244	1.520	2
0.099	1.537	3
0.049	1.328	4
0.024	1.204	5
0.009	0.633	6

These experiments show that the maximum rate of dissolution of the silver was obtained with a solution containing 0.10 per cent sodium cyanide.

RATES OF DISSOLUTION OF GOLD-SILVER ALLOYS

Dissolution tests in 0.10 per cent sodium cyanide solutions were run on two gold-silver alloys containing respectively 79.8 and 57.5 per cent gold. Fig. 7 shows the results. At the end of the tests the solutions were assayed for gold. The results, which are given in Table 4, show that the amounts of gold and silver dissolved are practically proportional to the composition of the alloys.

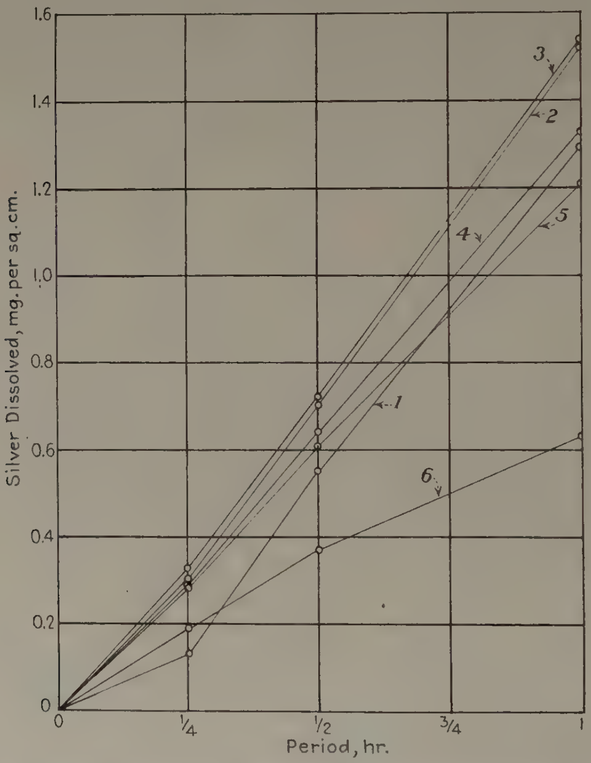

FIG. 6.—CUMULATIVE SILVER LOSS IN VARIOUS STRENGTHS OF CYANIDE.

TABLE 4.—*Relation between Composition of Alloys and Their Rate of Dissolution*

	Composition of Original Metal, Per Cent		Composition of Dissolved Metal, Per Cent	
	Gold	Silver	Gold	Silver
Alloy A	79.8	20.2	78.6	21.4
Alloy B	57.6	42.4	56.5	43.5

EFFECT OF ALKALINITY ON RATE OF DISSOLUTION OF GOLD

The effect of varying alkalinity on the rate of dissolution of gold in cyanide solutions was studied by the method described. A tube filled with asbestos impregnated with sodium hydroxide was used in the air line to remove any carbon dioxide. All tests were made in 0.10 per cent sodium cyanide solutions. Varying amounts of lime water or sodium hydroxide solution were added to produce the alkalinity desired. The

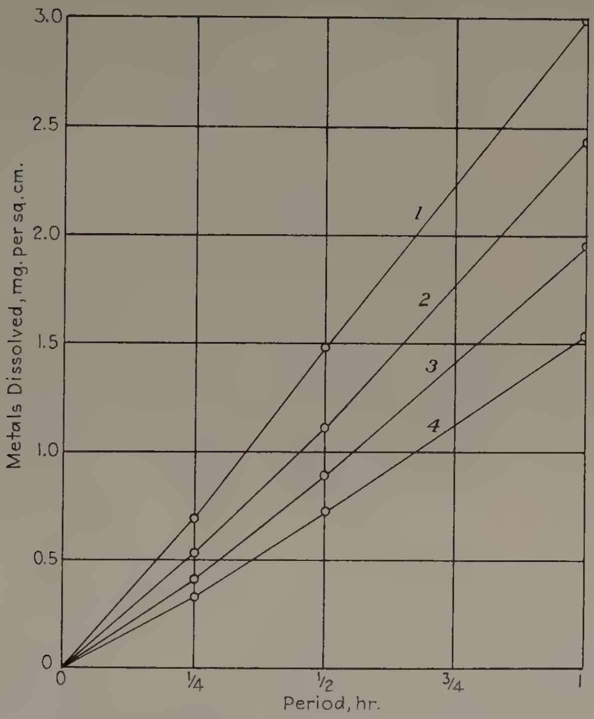

FIG. 7.—CUMULATIVE LOSS OF GOLD, SILVER AND GOLD-SILVER ALLOYS IN 0.10 PER CENT NaCN.

	Gold, Per Cent	Silver, Per Cent
1.	100.	
2.	79.8	20.2
3.	57.6	42.4
4.		100.

TABLE 5.—*Rates of Dissolution with Solutions Containing Lime*

CaO, Per Cent	pH	Average Rate of Dissolution First 15 Min., Mg. per Sq. Cm. per Hr.	Refer to Fig. 8, Curve No.
0.00	10.3	2.892	1
0.002	10.7	2.600	
0.0045	11.6	0.720	
0.008	11.7	0.440	2
0.0085	11.7	0.420	
0.011	11.8	0.075	3
0.024	12.1	0.050	4
0.047	12.2	0.025	5
0.110	12.6	0.025	6

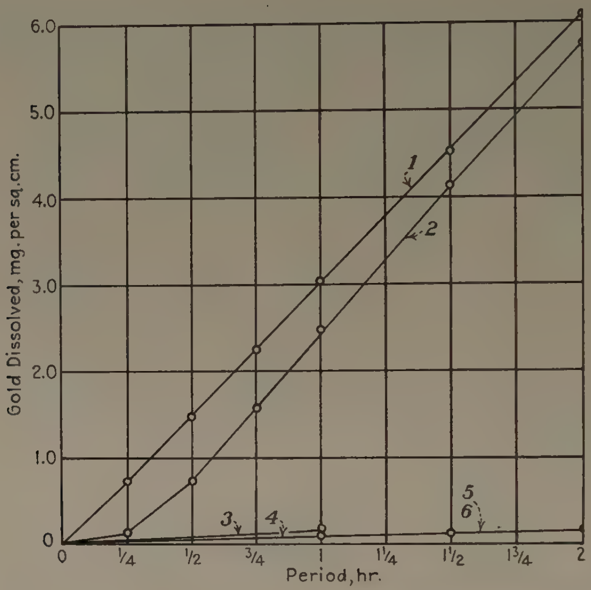

FIG. 8.—CUMULATIVE GOLD LOSS IN 0.10 PER CENT NACN SOLUTIONS CONTAINING VARYING AMOUNTS OF LIME.

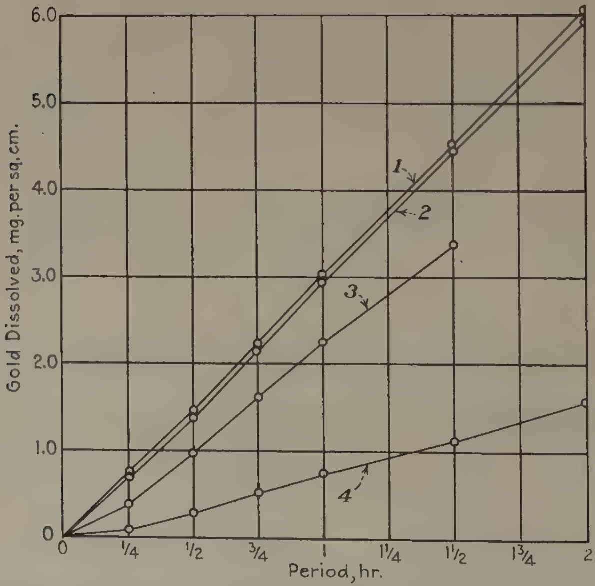

FIG. 9.—CUMULATIVE GOLD LOSS IN 0.10 PER CENT NACN SOLUTIONS CONTAINING VARYING AMOUNTS OF SODIUM HYDROXIDE.

solutions were analyzed for cyanide and alkalinity before and after each test.

Results obtained with cyanide solutions containing lime are shown in Fig. 8, those on solutions containing sodium hydroxide in Fig. 9.

The average rates of dissolution during the first 15 min. of the tests with the solutions containing lime are shown in Table 5, together with the pH values of the solutions. Table 6 gives similar data for the solutions containing sodium hydroxide.

TABLE 6.—*Rates of Dissolution with Solutions Containing Sodium Hydroxide*

NaOH, Per Cent	pH	Average Rate of Dissolution First 15 Min., Mg. per Sq. Cm. per Hr.	Refer to Fig. 9, Curve No.
0.00	10.3	2.892	1
0.07	12.0	2.804	
0.16	12.5	2.720	2
1.36	13.3	1.584	3
2.57	13.4	0.36	4

In Fig. 10 these average rates of dissolution have been plotted against the corresponding pH values of the solutions. As would be expected, the rate of dissolution is greatly reduced at high pH values; that is, at high concentrations of OH ions. The great difference in the shape of curves, however, shows that some other influence is at work, causing apparently a very pronounced reduction in the rate of dissolution in the solutions containing lime. Further experiments, described in the next section, were made to throw some light on the cause of this unexpected action of lime in cyanide solutions.

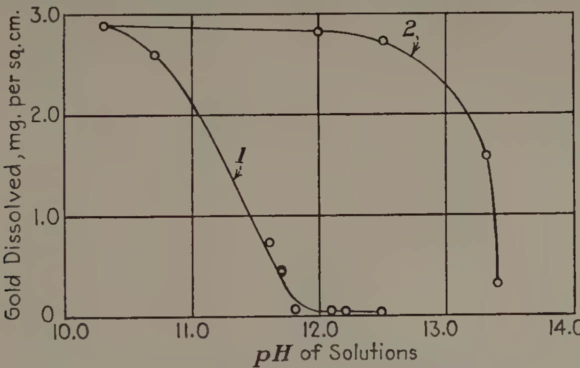

FIG. 10.—RATE OF DISSOLUTION OF GOLD IN 0.10 PER CENT NaCN SOLUTIONS OF VARYING ALKALINITY.

Curve 1, alkalinity due to lime; 2, alkalinity due to sodium hydroxide.

EFFECT OF CALCIUM COMPOUNDS ON DISSOLUTION OF GOLD IN CYANIDE SOLUTIONS

Lime.—Reduced solubility of oxygen in cyanide solutions containing lime was a possible explanation of the facts noted in the preceding paragraph. Determinations of the oxygen content of 0.10 per cent sodium cyanide solutions containing lime and saturated with air were therefore made by the method of Weinig and Bowen.(14) Table 7 gives the results. These figures show that lime has no appreciable influence on the solubility of oxygen in the cyanide solutions used.

TABLE 7.—*Solubility of Oxygen in* 0.10 *Per Cent NaCN Solutions Containing Lime*

CaO, PER CENT	OXYGEN, MG. PER LITER
0.00	8.20
0.045	8.63
0.105	8.57

Calcium Sulfate and Calcium Chloride.—To study the effect of calcium compounds other than lime, determinations of the rate of dissolution of gold were made in 0.10 per cent sodium cyanide solutions to which calcium sulfate and calcium chloride were added. It was found that 0.20 per cent calcium sulfate in solution had only a very slight retarding action. The effect of calcium chloride was more pronounced, but as much as 1.0 per cent in a 0.10 per cent sodium cyanide solution reduced the rate of dissolution only about 20 per cent, as compared with a reduction of around 98 per cent caused by 0.11 per cent of lime. In a calcium cyanide solution, prepared by treating lime water with hydrogen cyanide and containing 0.094 per cent $Ca(CN)_2$, the rate of dissolution was found to be very slightly less than in a sodium cyanide solution of equivalent cyanide content. No alkali was added to any of these solutions, so their pH values ranged from 10.1 to 10.4, all very close to the value of 10.3 found for 0.10 per cent sodium cyanide.

From these results it is evident that the great reduction in the rate of dissolution of gold in sodium cyanide solutions caused by the addition of lime is due neither to lower solubility of oxygen in the solutions nor simply to the presence of calcium ions. Apparently, both calcium and hydroxyl ions must be present to produce the full effect.

An adequate explanation of this point, as well as of several others brought out in this work, cannot be given at present because the necessary data are not available. It seems probable that the explanation lies in the effect of some of the substances present in one or more of the intermediate steps in the reaction between gold and cyanide, but this is of course merely a guess. The authors expect to continue the investigation with the object of clearing up some of these fundamentals in the chemistry of cyanidation.

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Milling Gold Ores at Pioneer

By Russell Spry,* Member A.I.M.E., and Paul Schutz†

(Spokane Meeting, September, 1934)

The property of Pioneer Gold Mines of B.C. Limited is in the Lillooet mining district of British Columbia. It is on Cadwalleder Creek, 54 miles distant by motor road from Shalalth, a station on the Pacific Great Eastern Railway.

Interests identified with the present company installed a small amalgamation plant on the property in 1924, consisting of a jaw crusher, Bryan mill and amalgamation plates, in which 75 per cent extraction was made on mill feed averaging 1.0 oz. gold per ton, the tailing being stacked for future treatment.

That plant was operated until 1928, when the present company was formed and a 100-ton continuous countercurrent decantation cyanide plant was built. A second unit, having a nominal capacity of 200 tons daily, was added in 1932 and put into operation during September of that year. When handling ore comparatively free from slow-settling material, the combined capacity of the two units is 400 tons, the original plant now handling about 140 tons maximum.

Production to date, from Pioneer, has been about $6,500,000, the present monthly production being better than $250,000 from about 370 tons ore daily, the recovery being 97 per cent. During the fiscal year ending March 31, 1934, a total of 112,936 tons ore and old amalgamation tailing was milled, the mill feed averaging 0.7670 oz. gold and the recovery amounting to 0.7422 oz. gold per ton, or 96.77 per cent. Milling costs, including refining and mint charges, during this period, amounted to $1.45 per ton of ore.

Since the building of the first unit of the cyanide plant in 1928, direct cyanidation has been employed, no attempt being made to remove coarse gold from the circuit, other than that cleaned up from the grinding mills during relining and from the classifiers, as occasions permit.

In the hope of still further increasing recovery, researches are going forward at this time with the view to removing some of the sulfides after primary grinding, the expectation being that selective treatment of this more refractory portion of the ore may hold economic possibilities.

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THE ORE

The ore, as received at the mill, consists of quartz carrying free gold, with small amounts of pyrite, arsenopyrite and pyrrhotite with chalcopyrite, sphalerite, stibnite and marcasite present in smaller quantities. The sulfide minerals probably comprise about 2 per cent of the mill tonnage. Minor gangue minerals include calcite, ankerite, mariposite, scheelite, sericite, etc. Country rock is quartz diorite, altered to sericite near the vein. This sericite slimes readily and is unwelcome in the mill because of its slow-settling characteristic.

Tests on mill tailing have suggested the presence of telluride but no confirmation has been made of this guess. The quartz is of two types, clear and ribbon, the latter containing streaks of shattered sulfides, which are readily exposed to cyanide on grinding. The portion of the gold that is visible is usually found on the ribbon faces and in the quartz close to the ribbon.

CRUSHING

The ore is dumped over 5-in. grizzlies at various points underground, the oversize is sledged and the undersize is hoisted to the surface in 2-ton skips to a 70-ton receiving bin near the shaft collar. As each skip load is dumped, a lime feeder is automatically tripped, most of the make-up lime needed in the cyanide circuit being added at this point.

From the coarse-ore receiving bin, a roller feeder draws the ore on to a 24-in. seven-ply rubber conveyor belt, 25-ft. centers, which feeds the crusher. The feeder is operated by a ratchet from the conveyor drive. Protecting the crusher from tramp steel is an Ohio magnet, 29 in. in diameter, located above the head pulley of the conveyor. The magnet is served by a Fairbanks direct-current generator developing 5 kw. at 115 volts and 43.5 amp. and operating at 1300 r.p.m. The generator is operated by the same motor that drives the crusher.

The conveyor discharges the ore on to a 4 by 5-ft. Hum-mer screen, type 70, set for ½-in. product on a slope of 36°. The vibrators are excited by a 0.5-kw. generator developing 11.4 amp. at 110 volts. This 15-cycle generator is driven at a speed of 865 r.p.m. by a 1½-hp. motor to which it is directly connected. Tyler Tyrod screen No. 9304, having apertures ⅞ by 5 in. is used. Each screen is turned end for end before being discarded, screen life being equal to about 23,000 tons of ore. Single-stage crushing is practiced at Pioneer, the Hum-mer oversize being crushed in a 3-ft. Traylor bell-head reduction crusher, No. 37, type TZ. The crusher is driven through a Texrope drive, by a 75-hp., 1145-r.p.m. slip-ring motor. The crusher is set for ½-in. product, the crushed product, together with the Hum-mer screen undersize, being transported on a 21-in. rubber conveyor belt, 50-ft. centers, to a storage bin above the upper terminal of a two-bucket aerial tram, which serves the mill, about 300 ft. away.

On the basis of 400 tons ore milled in 24 hr., the crusher is operated during two shifts daily, crushing but a portion of this time. The concave ring, which is of one piece in this type of crusher, is good for 95,000 tons service, while the mantle is changed after crushing 90,000 tons of ore. Of the ore hoisted from the mine, about 40 per cent bypasses the crusher as screen undersize. The sizes of Hum-mer screen undersize, crusher discharge and mill feed are given in Table 1. In order to operate the crusher to best advantage, consideration is being given to operating it in closed circuit with a second screen.

TABLE 1.—*Undersize, Crusher Discharge and Mill Feed*

Screen Opening	Hum-mer Undersize, Per Cent		Crusher Discharge, Per Cent		Mill Feed, Per Cent	
	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
Inch						
+⅞	1.5	1.5	12.2	12.2	6.9	6.9
+⅝	4.9	6.4	27.8	40.0	7.7	14.6
+¼	41.0	47.4	25.5	65.5	34.2	48.8
Mesh						
+6	21.5	68.9	14.0	79.5	16.2	65.0
+10	20.5	89.4	7.4	86.9	13.1	78.1
+20	10.4	99.8	4.5	91.4	9.6	87.7
+28	0.1	99.9	2.0	93.4	5.3	93.0
−28	0.1		6.6		6.6	

Mill feed is sampled automatically by a Geary-Jennings sampler below the head pulley of the conveyor that handles the crusher product and Hum-mer screen undersize. This sampler cuts the ore stream every 2½ min., the sample amounting to 500 lb. each day. This is coarse-riffled, crushed to minus ⅜ in. and fine-riffled to a 50-lb. sample, which is sent to the assay office for further reduction before assaying.

GRINDING AND CLASSIFICATION

Two cylindrical bins, built of fir, are used for fine ore. That serving the original mill unit is 14 ft. in diameter by 40 ft. high, having a capacity of 400 tons. That serving the newer mill unit measures 17 ft. in diameter, is also 40 ft. high and holds about 500 tons.

Grinding in cyanide solution is carried out in two stages. In the first unit, the primary mill, an Allis Chalmers ball mill, 5 by 4 ft., is fed by a 16-in. rubber conveyor belt equipped with adjustable ratchet drive on the head pulley. This mill is in direct closed circuit with a Dorr classifier, type DSC-20, 4 ft. 6 in. by 21 ft. 8 in. Classifier overflow is laundered to a 2-in. Wilfley pump, which raises it, together with the secondary mill discharge, to the secondary classifier—a Dorr bowl classifier, type DSDB, 6 by 30 by 10-ft. dia. The secondary mill is an Allis-Chalmers ball mill, 4 by 16 feet.

The primary mill in this first unit operates at 28 r.p.m. while the speed of the secondary is 30 r.p.m. Ball loads are 6000 and 11,000 lb. respectively. The primary mill, feeder and primary classifier are driven by a 24-in. Tutthill water wheel, with a 50-hp. squirrel-cage high-torque motor as a stand-by. The secondary mill is driven by a 36-in. Tutthill water wheel, with a 125-hp. slip-ring motor in reserve. The primary classifier, on a slope of 3 in. per foot, operates at 24 r.p.m., while the bowl classifier, in closed circuit with the secondary mill, on the usual 2-in. slope, has a speed of 23 r.p.m. in the reciprocating rake compartment and 4½ r.p.m. in the bowl.

In the newer unit of the plant, the primary grinding mill, a 64½-in. Marcy ball mill, fed by a 21-in. rubber conveyor belt, is operated in direct closed circuit with a Dorr classifier, type DSF, 8 ft. by 23 ft. 4 in. The overflow of this classifier is laundered to a 4-in. Wilfley and, with the discharge from the secondary mill, raised to the secondary classifier, a Dorr bowl, type DSFB, 8 by 30 ft. by 12-ft. dia. This bowl classifier is in closed circuit with the secondary mill, a 6 by 7 ft. Traylor ball mill. The Marcy mill, driven by a 100-hp. slip-ring motor, operates at 26 r.p.m. while the Traylor mill, driven by a 150-hp. slip-ring motor, operates at 24 r.p.m. Texrope drives are used on both mills. The ball load in the primary is 10,000 lb.; that in the secondary, 17,000 lb.

The primary classifier, in this unit, on a slope of 3⅝ in., has a speed of 28 r.p.m. It is driven by a 7½-hp. motor mounted on its tank and is equipped with a motor-driven chip remover, also mounted on the tank. The bowl classifier, on a slope of 2 in., has a speed of 26 r.p.m. in the reciprocating compartment, while the bowl rakes have a speed of 4 r.p.m. A 10-hp. motor, mounted on this classifier, drives both sets of rakes. Primary classifiers, baffled at the overflow ends to permit coarse separations, overflow at a few per cent on 10 mesh. The bowl classifiers, overflowing the finished product, deliver to the primary thickener pulps containing from 65 to 75 per cent minus 200 mesh. These finished overflows will contain from 2 to 5 per cent plus 100-mesh material, with the density at 18 per cent solids.

Primary ball mills are operated at a density of 71 per cent while about 67 per cent solids is the density in the secondary mills. In the former, 5-in. forged-steel balls are added daily, the consumption being 1.48 lb. per ton of ore ground. In the secondary mills, 1½ and 2-in. balls are added daily, the consumption being 2.23 lb. per ton, making a total ball consumption of 3.71 lb. Liner consumption amounts to 0.95 lb. per ton, being made up of 0.56 lb. for primary and 0.39 lb. for secondary

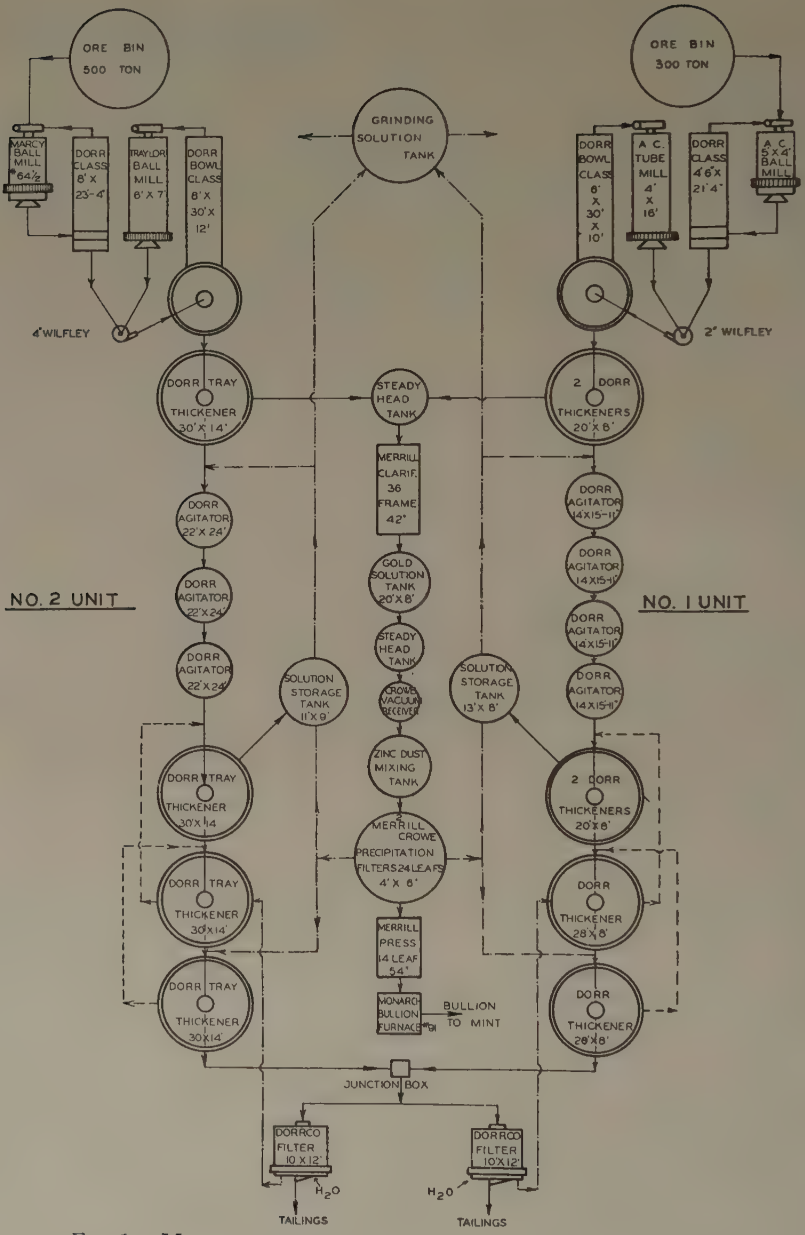
ORE BIN 500 TON
ORE BIN 300 TON
GRINDING SOLUTION TANK
MARCY BALL MILL #64½
DORR CLASS 8' X 23'-4'
TRAYLOR BALL MILL 6' X 7'
DORR BOWL CLASS 8' X 30' X 12'
DORR BOWL CLASS 6' X 30' X 10'
A C. TUBE MILL 4' X 16'
DORR CLASS 4'6" X 21'4"
A C. 5' X 4' BALL MILL
4" WILFLEY
2" WILFLEY
DORR TRAY THICKENER 30' X 14'
STEADY HEAD TANK
2 DORR THICKENERS 20' X 8'
MERRILL CLARIF. 36 FRAME 42"
DORR AGITATOR 22' X 24'
DORR AGITATOR 22' X 24'
DORR AGITATOR 22' X 24'
DORR AGITATOR 14' X 15'-11"
DORR AGITATOR 14' X 15'-11"
DORR AGITATOR 14' X 15'-11"
DORR AGITATOR 14' X 15'-11"
NO. 2 UNIT
NO. 1 UNIT
GOLD SOLUTION TANK 20' X 8'
STEADY HEAD TANK
CROWE VACUUM RECEIVER
SOLUTION STORAGE TANK 11' X 9'
SOLUTION STORAGE TANK 13' X 8'
ZINC DUST MIXING TANK
DORR TRAY THICKENER 30' X 14'
2 DORR THICKENERS 20' X 8'
2 MERRILL CROWE PRECIPITATION FILTERS 24 LEAFS 4' X 6'
DORR TRAY THICKENER 30' X 14'
DORR THICKENER 28' X 8'
MERRILL PRESS 14 LEAF 54"
DORR TRAY THICKENER 30' X 14'
DORR THICKENER 28' X 8'
MONARCH BULLION FURNACE #91
BULLION TO MINT
JUNCTION BOX
DORRCO FILTER 10' X 12'
DORRCO FILTER 10' X 12'
H_2O
H_2O
TAILINGS
TAILINGS

Fig. 1.—Mill flow sheet, Pioneer Gold Mines of B. C. Ltd.

grinding. Manganese-steel liners are used in the primary mills and chrome-steel in the secondaries. This rather high liner consumption is partly due to variations in mill speed caused by variations in electrical frequency, this condition having been overcome recently.

With a view to effecting economies in grinding, rubber shell liners have recently been installed in the 6 by 7-ft. Traylor mill, the intention being to use similar liners in the other secondary mill if the rubber is satisfactory. These liners are of 1½-in. rubber, backed by two-ply 32-oz. duck, and are held in place by manganese-steel retaining bars bolted to the shell.

The 2-in. Wilfley pump, which handles the primary classifier overflow and secondary mill discharge, in the first unit, is belt-driven at 865 r.p.m. It operates under a total head of 20 ft., the specific gravity of the pulp being 1.4. In this pump a runner lasts 28 days while the life of a casing is from 40 to 55 days. In the second unit, the 4-in. Wilfley, performing similar service, is directly connected to a 25-hp. motor, operates at 865 r.p.m. under a head of 45 ft. and handles a pulp of 1.35 sp. gr. Runner life in this instance is 11 to 16 days, casing life 30 to 37 days, and 60 days for a die ring. Rubber follower plates last 3 and 2 years on the 2-in. and 4-in. pumps, respectively.

Dorr classifiers, at Pioneer, are operated primarily as classifiers, rather than as sizers. Particularly is this true in regard to the bowl classifiers where advantage is taken of their ability to retain the heavy sulfides in the circuit, until they are ground much finer than the lighter quartz particles. The selective grinding of sulfides at Pioneer, made possible through bowl classification, is an important factor in reducing tailing loss. Evidence of the concentration of values in the bowl-classifier rake product was noted in a recent test. Assays of bowl sands were about four times those of original mill feed, despite the fact that about 70 per cent of the original values had been dissolved in the grinding and classification circuits.

Agitation and Thickening

Bowl-classifier overflows are laundered to the primary thickeners. In the first unit two Dorr type R thickeners, 20 by 8 ft., operating in multiple, are used for decantation of the pregnant solution. Continuous countercurrent washing of the residue, following agitation, is carried out in three steps, two 20 by 8-ft. Dorr thickeners being used for the first wash, with the second and third wash being made in two single 28 by 8-ft. type R thickeners. The rakes in these thickeners are operated at a peripheral speed of 12 to 15 ft. per minute.

Agitation in this first unit is carried out, between the primary and secondary thickeners, in a series of four Dorr agitators, type S, 14 ft. by 15 ft. 11 in., the rake speed being 5½ r.p.m. In the newer mill unit, four

Dorr balanced tray thickeners, type RTB, 30 by 14 ft., are used, one as a primary, three as secondary thickeners, for washing the residue after agitation. In this unit, agitation is conducted in three Dorr agitators, type H-D, 24 by 22 ft., operating in series, the rake speed being 5 r.p.m. Each of these larger agitators is supplied with 78 cu. ft. free air per minute at 30 lb. pressure; density of pulp during agitation is between 50 and 52 per cent while thickener underflows are held as closely as possible to 50 per cent solids.

On the basis of 400 tons ore being milled daily, in the combined plant, the thickener area provided amounts to 4.9 sq. ft. per ton per 24 hr. Agitation capacity is 44 hr. retention, based on the same tonnage. Thickener underflows are handled, in each case, by Dorrco diaphragm suction pumps, 4-in. simplex pumps being used in the first unit while 4-in. duplex pumps are used in the newer plant. Because of the necessity of greater elevation between successive thickeners, in a countercurrent decantation plant, where balanced tray thickeners are used, compared to single compartment thickeners, Inspiration-type Dorrco diaphragm suction pumps were installed for transferring thickener underflows in two of the washing steps at Pioneer. In this Inspiration-type pump, the usual solid eccentric rod is replaced by a 4-in. pipe, the pipe acting as an eccentric rod and at the same time as a discharge tube through which the pulp is lifted to a height of 5½ ft. above the pump bowl.

Diaphragm pumps at Pioneer are operated at 66 r.p.m. and at fairly short stroke. This results in unusually long diaphragm life at this plant, 18 months to 3½ years being the life of diaphragms on the regular Dorrco pumps, with 22 months on the Inspiration-type pumps.

Repulping thickener underflows with wash solution is done in air-lifts, in the first mill unit. In the newer plant repulping is conducted in Dorrco repulpers, 15 by 15 in. by 8 ft. The mechanical mixing of residue and wash solution is more thorough and is credited with contributing to the better performance of the second unit.

Based on mill heads averaging 0.7133 oz. gold per ton for the first five months of 1934, dissolution from daily assays is as shown in Table 2. This table shows that 95.59 per cent of the gold is in solution at the end of agitation. More correctly stated, this amount was dissolved or removed from the circuit before the pulp was fed to the secondary thickeners. Of the gold produced at the Pioneer mill, about 4 per cent of the total is recovered from ball mills and classifiers, these recoveries being made when the mills are relined. Although the grind in the older unit is consistently finer than that in the newer unit, there is only 61 per cent of the gold values dissolved or tied up, prior to agitation, in the former unit, compared to 69 per cent in the latter. This is due perhaps to the fact that higher circulating loads are carried in the grinding sections of the newer unit, resulting in longer contact with solution.

TABLE 2.—*Dissolution of Gold*

	Assay (Solids) Oz. Gold per Ton	Gold Dissolved, Per Cent	
		Ind.	Cum.
Mill feed	0.7133		
Agitator feed	0.2405	66.28	66.28 prior to agitators
Agitator discharge	0.0324	29.31	95.59 in agitators
No. 2 thickener underflow	0.0227	1.23	96.82 in No. 2 thickener
No. 3 thickener underflow	0.0207	0.27	97.09 in No. 3 thickener
No. 4 thickener underflow	0.0199	0.12	97.21 in No. 4 thickener
Filter cake to waste	0.0189	0.14	97.35 in filter

The six thickeners, six simplex diaphragm pumps and four agitators in the first unit are driven by a 25-hp. motor through a line shaft. In the second unit the four two-compartment tray thickeners, four duplex diaphragm pumps, and three repulpers are driven from a line shaft powered by a 25-hp. motor through a double-reduction Gilmer multi-V-belt drive. The three agitators are driven by a 20-hp. motor through a similar type of drive.

While grinding, classification, agitation and thickening are carried out in two units, at Pioneer, common filtration, clarification and precipitation systems serve both mill sections.

FILTRATION AND DISPOSAL OF TAILING

Final thickener underflow in No. 1 unit is withdrawn by a diaphragm pump, laundered to a sump, and elevated by a 2-in. Wilfley pump to a steady-head box. The underflow of the final thickener in No. 2 unit is laundered to this same box, the combined underflows being split to two Dorrco washing filters, each 10 by 12 ft. Each filter is driven by a 7½-hp. motor through a speed reducer and roller chain drive mounted on the trunnion end of the filter, the filter speed being ⅕ r.p.m. One Ingersoll-Rand dry vacuum pump, type ER-1, 22 by 9-in., driven at 275 r.p.m. by a 40-hp. motor through a Texrope drive, serves the two filters. A second pump of the same size is held in reserve. This amounts to vacuum pump displacement of 1.45 cu. ft. per min. per sq. ft. of canvas.

Cake discharge is aided by air at 2 lb. pressure, provided by a No. 25-B Connersville blower for each filter. Each blower is directly connected to a 5-hp. motor. The filtrate from each unit is handled by a Gould 1½-in. centrifugal pump, submerged in solution, and driven by a 3-hp. motor.

The filter cloth used is No. 26 twill. It is backed by cider-press cloth, both the twill and the backing being caulked into place in panel grooves with manilla rope. With cake thickness of ½-in., a vacuum of 22 to 23 in. is maintained, the moisture of the washed cake being 18 to 19 per cent.

The amount of water used in the sprays is recorded by meters. Washed filter cake is removed from the filters by screw conveyors, because of the sticky nature of the cake, and repulped with water prior to being conveyed through 3000 ft. of machine-banded pipe to a point below the water intake of Bralorne mines, where it is discharged into Cadwalleder Creek.

Filter cloth is treated with acid every 21 days, this procedure consisting of hosing out the filter, dumping in a dilute solution of hydrochloric acid and rotating the filter drum for an hour; the filter is then drained, again hosed out and put into service. There is no scrubbing of the cloth, the pores being easily kept open throughout the cloth life. To keep the cloth in best condition, low-pressure air is blown through it for several seconds after the cake has been discharged. Changing a filter cloth, which is done after about 108 days service, requires two men for about 8 hr., the time being counted from the shutting off of the filter feed until the machine is placed in service again. Each filter has 371 sq. ft. of canvas area, which, on the basis of washing 200 tons of solids per day, shows a capacity of 1078 lb. per square foot of canvas per 24 hours.

Filtrate is pumped to the second last thickeners, in the washing series, although this procedure is varied at times, depending on the values in the filtrates. Dissolved loss, discharged with filter cake, for the first five months of 1934 has averaged 0.0023 oz. gold per ton of ore, while dissolution on the filter has been 0.001 oz. gold per ton. During the six-month period preceding, the dissolution on the filters amounted to 0.004 oz. gold per ton of ore.

Clarification and Precipitation

Pregnant solution, amounting to approximately 3.25 tons per ton of ore, overflowing the primary thickeners, is pumped from No. 1 unit to a 6 by 8 ft. steady-head tank. From No. 2 unit the primary thickener overflow flows by gravity to the same tank, where lead acetate is added. From the steady-head tank the unclarified pregnant solution is pumped, at 15 lb. pressure, by a 3-in. Krogh pump directly connected to a 15-hp. motor, through a Merrill center-sluicing clarifying filter consisting of 36 frames, each 42 in. square. Each plate is covered with 10-oz. duck. The normal life of the duck covers is 31 days, the life being shortened if colloidal material, to any marked extent, is permitted to overflow the primary thickeners. No acid wash is given these filter cloths, minute holes appearing before there is any marked coating of lime. The press is sluiced twice a shift with water at 60 lb. pressure, a 16 by 15 ft. storage tank being provided to handle the unclarified solution while the filter is being sluiced.

Clarified solution flows by gravity from the Merrill filter to a 20 by 8-ft. storage tank. The solution is heated in this tank, by means of an

8-in. header in the tank, through which mine air is conveyed from the compressors to the receivers. The writers believe that in this plant there is an advantage of maintaining the temperature of the pregnant solution above 60° F. during precipitation. From the gold-solution storage tank, flow is by gravity to a 6 by 5-ft. steady-head tank ahead of the Crowe vacuum tank where an 8 by 5-in. Ingersoll-Rand air-cooled vacuum pump, driven by a 3-hp. motor and Texrope drive, is used to remove oxygen from the solution. A 3-in. Krogh pump, submerged in the steady-head tank, and driven by a 7½-hp. motor, elevates the solution to a 3 by 10-ft. mixing tank, zinc dust (Merrillite) being added at the rate of 0.06 lb. per ton of solution. An auger-type feeder is used for introducing the zinc dust.

From the mixing tank the solution is pumped by a 3-in. centrifugal, directly connected to a 7½-hp. motor, to a Merrill-Crowe low-level vacuum leaf filter, where precipitation is completed. This filter consists of twenty-four 4 by 6 ft. leaves in a 10½ by 12-ft. wood tank, an impeller operating at 175 r.p.m. providing the necessary agitation. Each filter leaf is covered with burlap, over which are two pequot sheeting bags. The life of the burlap is one year, while the sheeting bags last about 2½ months. A clean-up is made every 8 days, the time required being about 2 hr. for two men. Two Merrill filter tanks are provided, one being placed in service before the second is withdrawn for the clean-up. In this way, there is no interruption in the flow of solutions and cleaning up can be done more thoroughly.

In cleaning a filter, precipitate is hosed off the leaves by pregnant solution and pumped to a press in the refinery. After a month's service on the precipitation filter, the outer sheeting bags are removed and burned in the refinery. New bags are installed as inner bags, and the old inner bags, after acid treatment, are used as outer bags.

Pregnant solution, averaging 0.2164 oz. gold, is precipitated to about 0.002 oz. gold, the resulting barren solution being pumped to the final thickeners in the washing series, by a 3-in. Krogh pump directly connected to a 10-hp. motor. Tonnage of solution precipitated was formerly figured by means of weir boxes but is now recorded by a Tagliabue flowmeter. The installation of the latter has been too recent to permit of detailed comment at this time.

Refining

The Merrill filter press in the refinery, into which the precipitate is pumped, consists of fourteen 54-in. triangular leaves. Each leaf is covered with one sheet of kraft paper and one thickness of pequot sheeting. The paper is used once and then burned, while the sheeting life is 4 months, after which time it also is burned. After the precipitate is pumped from the Merrill-Crowe precipitating filter to the press in the refinery, the refinery press is permitted to drain to a sump. The resulting solution is pumped back to the gold-solution tank. Low-pressure air is blown through the press for 48 hr., at the end of which time the precipitate contains 8 per cent moisture. The precipitate contains about 20.2 per cent gold and 3.8 per cent silver.

A Monarch Simplex furnace, 3 by 4 ft. inside the steel shell, is used for smelting and refining the precipitate. The furnace is lined with 6 in. of Carbofrax, giving inside lining measurements 2 by 3 ft. The life of a lining is from 110 to 130 hr. To reline the furnace takes three men about 14 hours. The bullion furnace is a Monarch tilting crucible furnace. The furnace is No. 125, but No. 80 graphite crucibles are used in it. Crucible life is about 192 hr., which corresponds to eight clean-ups. With both furnaces running there is used about 100 gal. diesel oil per 12 hr. Air is furnished by a Connersville low-pressure blower.

In smelting the precipitate, 10 batches, consisting of 20 lb. precipitate and 30 lb. flux each, are charged to the furnace. The 500-lb. charge is melted, the slag poured off and resulting gold buttons of 830 to 840 total fineness refined in the same furnace. In the refining operation, not over 60 lb. of bullion is charged at a time, the total fineness of the refined buttons being 925 to 935, of which 815 to 820 is gold. The refined buttons are melted in the bullion furnace and poured into bricks of about 700 oz. The resulting bricks are sampled by drilling three $\frac{3}{16}$-in. holes at the center and two corners, weighed, cleaned and shipped by mail to the mint at Ottawa in cardboard boxes.

Handling of Solutions

Overflows from the first secondary thickeners flow to two tanks, 13 by 8 ft. and 11 by 9 ft. respectively, this grinding solution being pumped to a 16 by 10-ft. storage tank above the ball mills, for use in the mills and as make-up in the classifiers. Barren solution can be diverted to the same storage if desired at any time.

Cassell brand sodium cyanide, 97 to 98 per cent NaCN, is added at the grinding mills and in the first two agitators in each unit, pregnant solution being kept at 1.40 lb. NaCN per ton solution, while grinding solution will be about 1.30 lb. Cyanide consumption for the first five months of the present year has averaged 0.87 lb. NaCN per ton of ore treated. Quicklime is added at the storage bin as the ore is dumped from the mine skips, also from drip boxes with feed to the first secondary thickeners. The amount of lime used is 4.23 lb. per ton of ore, an excess being desirable as an aid to settlement. Pregnant solution is kept at 2.36 lb. CaO per ton, while grinding solution is maintained at 2.1 lb. CaO.

Sampling and Assaying

While mill feed is automatically sampled, and the samples assayed daily, mill heads are calculated from bullion produced plus tailing, and recoveries are based on these heads. The mill heads, as determined by the automatic sampler and resulting assays, are consistently below the actual heads. Products into and out of each agitator series, each secondary thickener and each Dorrco filter, are sampled regularly and assayed daily, with the object of maintaining close control of operations. This procedure is very desirable because of the great variation in gold values in the feed, at times.

ACKNOWLEDGMENT

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Ore Treatment at Beattie Gold Mines Limited

By W. G. Hubler* and F. J. Martin,† Members A.I.M.E..

The Beattie mine is at about the center of Duparquet township in the Province of Quebec, Canada. It is 22 miles northwest of the town of Noranda. The elevation is approximately 1200 ft. above sea level. This locality, long considered a favorable one from the geological point of view, has been prospected for over 20 years, but no discoveries of economic importance were made until the present orebody was found by John Beattie in 1930.

The specific gravity of the ore is 2.77. The gold occurs in intimate association with the sulfides, pyrite and arsenopyrite, which in turn are very finely disseminated in a silicified porphyry. The bulk of the gold is associated with the pyrite, only a small proportion being with the arsenopyrite. There are small amounts of magnetite, ilmenite and chalcopyrite present in the ore. There is also some hematite in the ore that is mined from near the surface in the open pit.

Metallurgical Problems and Methods of Attack

1. The ore is extremely hard and resistant to grinding.
2. The sulfide minerals are finely disseminated in the gangue.
3. The gold, in turn, is so intimately associated with the sulfides that grinding to even 5 microns will not release all of the values for cyanidation.
4. It is necessary to make a high ratio of concentration in order to obtain a product sufficiently high in gold to meet high shipping and treatment charges.

Hardness of the Ore.—Owing to the extreme resistance of the ore to grinding, it was recognized that grinding costs would be high. Microscopic tests showed that grinding to 95 per cent minus 325 mesh would be necessary to liberate a sufficient amount of the values in order to obtain a tailing assaying 0.02 oz. gold per ton, which is considered to have the economical minimum gold content that can be obtained. It is not feasible to grind this hard low-grade ore (0.18 to 0.20 oz.) to 95 per cent minus 325 mesh, so it was necessary to overcome the problem in another way. The following method was decided upon: (1) Fine crushing to minus ¼ in.; (2) relatively coarse primary grinding; flotation of low-grade concentrates in the primary and rougher circuits and discarding the

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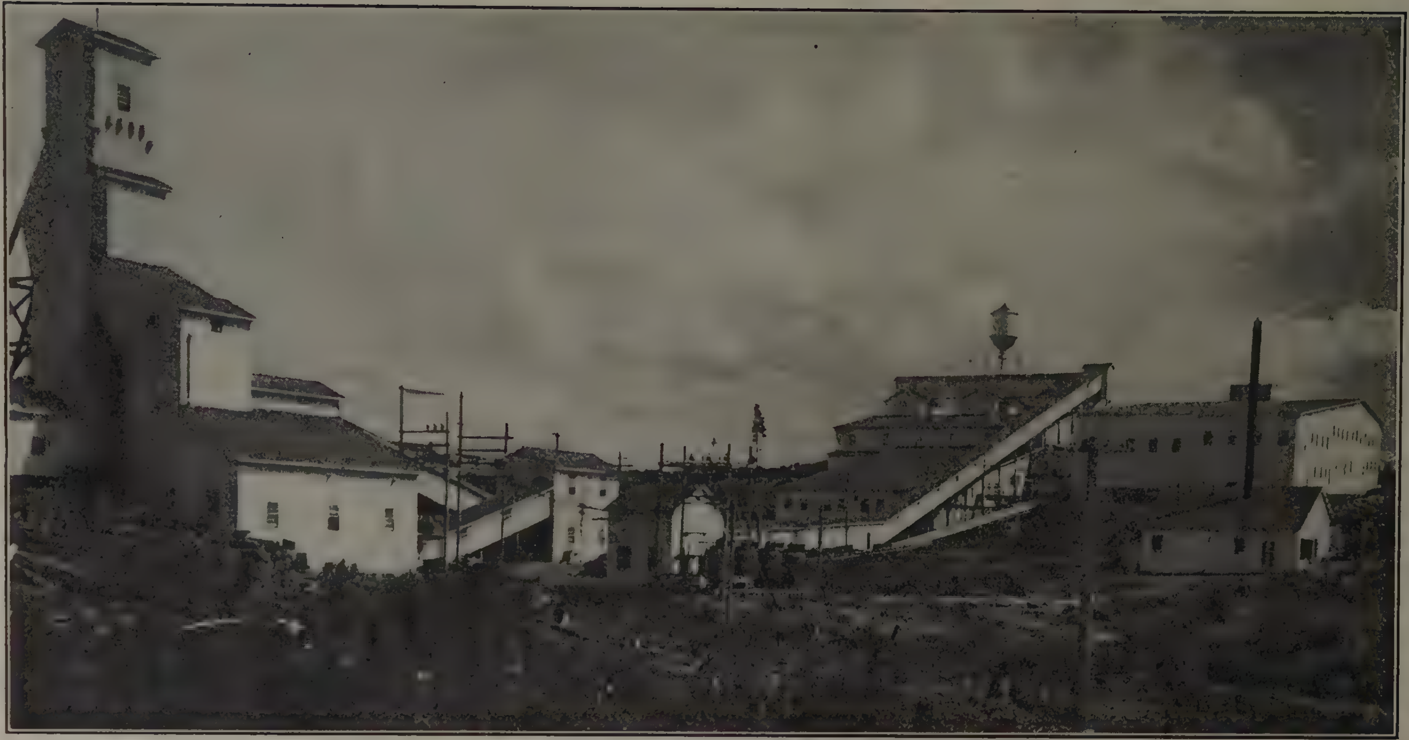

FIG. 1.—BEATTIE MILL, LOOKING SOUTH, SHOWING MAIN SHAFT, CRUSHER HOUSE, SCREEN HOUSE, FLOTATION MILL, NEW CYANIDE MILL, CENTRAL HEATING PLANT.
Filtering and drying building is immediately behind the ore bins.

bulk of material as a coarse tailing; (4) regrinding and refloating of the low-grade rougher concentrate discarding a tailing here also; (5) combining the concentrate obtained from the flotation of the reground rougher concentrate with the primary concentrate and regrinding both products to 95 per cent minus 325 mesh, then refloating this material to produce the final concentrate.

Table 1 gives the tonnages of products in the grinding and flotation circuits, and also some typical screen tests. The final tailing is discarded at 69 per cent minus 200 mesh, and the final concentrate is 94 per cent minus 325 mesh. By this three-stage method of grinding, only 15 to 16 tons out of every 100 tons milled are ground very fine, and this portion of the ore contains the sulfide minerals and offers less resistance to grinding than does the more siliceous gangue.

TABLE 1.—*Tonnages in Grinding and Flotation Circuits, with Typical Screen Tests*

TONNAGES[a]	TONS	TONNAGES	TONS
Feed to primary mills	100.00	Rougher concentrate	18.14
Feed to secondary mill	22.74	Final cleaner tailing	8.80
Feed to regrind mill	15.60	Primary rougher tailing	85.00
Final concentrate	7.00	Secondary tailing	8.00

SCREEN TESTS

Mesh	Primary Classifier Overflow, Per Cent	Secondary Classifier Overflow, Per Cent	Final Concentrate, Per Cent	Primary Rougher Tailing, Per Cent	Final Tailing, Per Cent
+ 60	1.5	nil	nil	2.0	1.0
+100	10.0	0.5	tr.	11.0	8.5
+200	23.5	15.0	1.5	24.5	21.5
+325	12.5	16.5	4.5	13.0	10.0
−325	52.5	68.0	94.0	49.5	59.0

[a] These figures include the circulating loads.

Dissemination of Sulfides in Gangue.—Table 2 was compiled from data that were obtained during a period when slightly coarser grinding was in practice than in the screen tests of Table 1, and it is included to illustrate the distribution of the grain sizes. This table shows that the bulk of the values occurs in the minus 325-mesh material. The intimate association of the sulfides is well illustrated in Fig. 2. The grain sizes are extremely

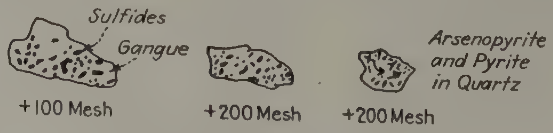

FIG. 2.—INDICATION OF INTIMATE ASSOCIATION OF SULFIDES IN QUARTZ GRAINS FROM CLASSIFIER OVERFLOW. × 450.

fine and doubtless they penetrate the gangue crystals as well as cover their surfaces.

TABLE 2.—*Dissemination of Sulfides in Gangue*

Mesh	Classifier Overflow, Weight Per Cent	Analysis, Per Cent Total Sulfides Present	Microscopic Examination Notes
+100	15.0	9.5	No free mineral.
+200	24.0	21.3	Approx. 5 per cent free mineral; 20 per cent middling and some hematite.
+325	9.3	15.2	Approx. 20 per cent free mineral; considerable hematite and iron-stained gangue.
−325	51.7	53.5	Approx. 80 per cent of sulfides free; remainder of sulfides fine middling.

In Fig. 3 are shown sketches of minus 200-mesh and minus 325-mesh grains from mill tails. These sketches show the impossibility of commercially grinding this middling fine enough to release the sulfide minerals.

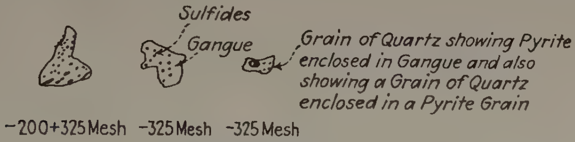
Sulfides
Gangue
Grain of Quartz showing Pyrite enclosed in Gangue and also showing a Grain of Quartz enclosed in a Pyrite Grain
−200+325 Mesh
−325 Mesh
−325 Mesh

FIG. 3.—INDICATION OF INTIMATE ASSOCIATION OF SULFIDES WITH QUARTZ IN GRAINS FROM MILL TAILS. × 450.

Table 3 gives an analysis of the distribution of minerals in the mill tailings, showing that the greater proportion of the tailing is really a fine middling product, only 40 per cent being ground free.

TABLE 3.—*Distribution of Minerals in Mill Tailings*

Mesh	Free Sulfides, Per Cent	Middlings, Sulfides Predominate, Per Cent	Middlings, Gangue Predominates, Per Cent	Free Gangue, Per Cent	Hematite, Per Cent	Pyrrhotite, Ilmenite, Per Cent
+100	nil	2.00	0.60	1.0	tr.	0.162
+200	nil	1.33	1.33	2.0	tr.	0.370
+325	nil	nil	0.94	5.0	1.0	0.168
−325	nil	tr.	53.30	27.0	2.0	1.325
	nil	3.33	56.17	35.0	3.0	2.025

Close Association of Gold with Sulfide Minerals.—The distribution of the sulfides and the distribution of the gold in relation to the sulfides are indicated in Table 4, showing that the recovery of the gold follows closely the recovery of the pyrite, and that the distribution of the gold agrees

TABLE 4.—*Screen Analyses of Composite Samples*

December 9 and 10, 1933

Mesh	Wt. Per Cent	Assay, Gold, Oz.	Gold Distrib., Per Cent	Assay, Iron, Per Cent	Assay, Sulfur, Per Cent	Arsenic, Per Cent	Arseno-pyrite, Calculated Per Cent	Sulfur		Iron not Pyrite or Arseno-pyrite, Per Cent	Pyrite, Per Cent	Recovery Pyrite Per Cent	Oz. Gold to Per Cent Pyrite
								As Arseno-pyrite, Per Cent	As Pyrite, Per Cent				
Heads:													
+100	15.0	0.15	10.1	3.25	2.265	0.30	0.65	0.12	2.145	1.61	4.02		0.037
−100 +200	24.0	0.21	22.6	4.20	3.157	0.42	0.91	0.17	2.987	1.28	5.60		0.037
−220 +325	9.3	0.28	11.7	5.40	4.119	0.56	1.21	0.23	3.889	1.59	7.29		0.038
−325	51.7	0.24	55.6	5.30	3.707	0.50	1.08	0.21	3.497	1.88	6.55		0.036
Heads	100.0	0.22	100.0	4.75	3.569	0.48	1.04	0.204	3.365	1.46	6.30		0.034
Concentrates:													
+100	tr.												
+200	2.4	1.06	1.1	18.90	21.00	2.91	6.32	1.24	19.76	1.54	37.05		0.028
−200 +325	10.0	1.78	7.4	27.20	29.93	4.00	8.69	1.70	28.23	2.25	52.93		0.033
−325	87.6	2.50	91.5	35.20	37.75	5.13	11.14	2.18	35.57	0.04	66.69		0.037
Conct.	100.0	2.39	100.0	34.00	36.65	5.00	10.86	2.13	34.52	0.07	64.72	85.0	0.036
											oz. Au	Loss in pyrite in tails	
Tailings:													
+100	13.3	0.058	20.3	2.05	0.824	0.30	0.65	0.12	0.714	1.22	1.31	0.045	0.044
−100 +200	22.0	0.045	26.1	2.20	0.515	0.18	0.39	0.076	0.439	1.69	0.82	0.036	0.054
−200 +325	9.7	0.040	10.2	2.15	0.412	0.15	0.32	0.06	0.352	1.74	0.65	0.026	0.061
−325	55.0	0.030	43.4	2.85	0.412	0.15	0.32	0.06	0.352	2.41	0.65	0.0195	0.046
Tailings:	100.0	0.038	100.0	2.45	0.549	0.20	0.43	0.08	0.469	2.04	0.87	0.033	0.043
								Oz. Gold to 1 Per Cent Pyrite Heads to Conct.,				0.035	

	Mesh	Gold		
		Oz.	Per Cent Recovery	
Heads	+100	2.25	68.8	Loss in tails other than pyrites, 0.01 oz. (probably hematite)
	−100 +200	5.04	81.8	
	−200 +325	2.604	86.2	
	−325	12.408	87.7	
		22.302	83.0	
Concentrates	+100	Tr.		
	−100 +200	0.196		
	−200 +325	1.377		
	−325	16.950		
		18.523		
Tails	+100	0.700		
	−100 +200	0.913		
	−200 +325	0.357		
	−325	1.522		
		3.492		

Heads, 100 tons; concentrates, 7.74 tons; tailings, 92.26 tons; ratio of concentration, 12.92 into 1.

closely with that of the pyrite. These facts indicate that the bulk of the gold occurs associated with the pyrite.

BEATTIE PRACTICE

Early metallurgical tests indicated that a 65 per cent recovery of the gold was all that could be obtained by cyanidation after grinding to 98 per cent minus 325 mesh. These tests also showed that this recovery would not be materially increased by finer grinding. Flotation tests indicated that after grinding 65 to 80 per cent to minus 200 mesh, a concentrate assaying 2.5 oz. gold per ton could be produced with a recovery between 83 and 87 per cent in this concentrate. A favorable smelting contract was offered on this material by the American Smelting and Refining Co., with the result that it was decided to erect a flotation concentrator and to market the concentrate until a more successful method for its treatment should be discovered. This concentrate is shipped to Tacoma, Wash., via Quebec and the Panama Canal. Our endeavor, therefore, is to preserve a high grade in this product, thereby reducing the shipping and smelting charges.

Crushing

The ore from the open pit is crushed to minus 10 in. in a 36 by 48-in. Buchanan jaw crusher which is installed underground. All development and part of the stope ore is hoisted without crushing at the present time, but when deeper development is completed, the crusher will be moved to a lower level and will be in a position to take the stope ore.

The ore is hoisted in 2½-ton skips and dumped into a 200-ton coarse-ore bin. From this bin the ore is fed over a roll grizzly feeder to a 30 by 42-in. Buchanan jaw crusher, which is set at 4-in. opening. The crusher discharge, together with the roll grizzly undersize, is conveyed by a 24-in. belt operated at 300 ft. per minute, to two 4 by 8-ft. double-deck Niagara screens. These screens have 1⅛-in. square opening, ⅜-in. wire on the top decks and ¼ by 4-in. opening on the bottom decks. The undersize from these screens is carried by a 24-in. conveyor operating at 250 ft. per minute to the mill ore bins. A Merrick weightometer is installed on this belt. The oversize from these screens is returned to the crushing plant on a 24-in. conveyor operating at 300 ft. per minute. This material is passed over a 4 by 6-ft. single-deck Niagara screen with 1-in. square openings and ⅜-in. wire. The oversize from the 4 by 6-ft. screen goes to a 30-ton surge bin and is fed by a 30-in. conveyor to a 4-ft. Symons standard cone crusher. The undersize goes to a 30-ton surge bin, from which it is fed to a 4-ft. Symons short-head cone crusher. The standard crusher is set at ¾ in., and the short-head crusher at ¼ in. The discharges of both cone crushers are carried to the screenhouse along with the jaw-crusher product, and both cone crushers are kept in closed circuit with the screens.

The crushing plant is compact, all crushers are in one building, and labor costs are small. Two men per shift operate the crushing plant, screens and conveyors; one operator for the three crushers and one for the screens and conveyors.

The capacity of the crushing plant is 90 tons per hour. The circulating load is 250 per cent, and the efficiency of the screens is about 75 per cent. The installed horsepower is 500, but the normal working load is 360 hp. The ore is resistant to crushing and the wear on manganese jaw plates and cone-crusher liners is excessive.

TABLE 5.—*Distribution of Crushing Cost*

	Crushing	Conveying	Total
Labor[a]	$0.0187	$	$0.0187
Supplies and maintenance	0.0373	0.0084	0.0457
Depreciation[b]	0.0172	0.0120	0.0292
Power	0.0228	0.0067	0.0295
Other costs	0.0035		0.0035
Totals	$0.0995	$0.0271	$0.1266

[a] Includes labor conveying.

[b] Includes write-off of conveyors in one year; and after one year's service they show very little wear.

TABLE 6.—*Typical Screen Tests on Crusher-plant Products*

Mesh		Discharges of Three Crushers (Screen Feed), Per Cent	Screen Oversize, Per Cent	Standard Symons Feed, Per Cent	Short-head Symons Feed, Per Cent	Screen Undersize Finished Product, Per Cent
Inch						
−4	+1⅛	32.8	50.4	77.3	nil	nil
−1⅛	+1	1.6	1.8	2.7	nil	nil
−1	+⅜	18.9	24.4	16.5	32.9	nil
−⅜	+¼	7.5	9.6	2.2	26.3	nil
−¼ in.	+10 mesh	39.2	13.8	1.3	40.8	81.6
Mesh						
−10	+20	nil	nil	nil	nil	6.2
−20	+35	nil	nil	nil	nil	3.2
−35	+60	nil	nil	nil	nil	1.7
−60	+100	nil	nil	nil	nil	1.2
−100	+200	nil	nil	nil	nil	1.1
−200	+325	nil	nil	nil	nil	0.6
	−325	nil	nil	nil	nil	4.4

Screen efficiency, 75.3 per cent; circulating load, 234 per cent.

Crushing costs for reducing run of mine ore from minus 20 in. to minus ¼ in., and conveying it to the mill bins, was 12.66¢ per ton of ore. This cost was distributed as shown in Table 5.

Some results of sizing tests on the products of the crushing plant are given in Table 6. The ore breaks in thin flat sections and this accounts for the high circulating load and the low efficiency of the screens. The finished product is characterized by the absence of fines. When crushing to minus ¼ in., it is not uncommon with other ores to produce 20 per cent or more of minus 100-mesh material; the Beattie ore, however, yields but 5 or 6 per cent of minus 100-mesh material. No fines are present in the run of mine ore.

Grinding and Flotation

The crushed ore is stored in three mill ore bins, which have a combined capacity of 2400 tons. The ore is fed from the bins by 30-in. belt conveyors to the primary ball mills, which consist of an 8-ft. by 72-in. Hardinge ball mill in closed circuit with a 12 by 28-ft. model D Dorr classifier, and a 10-ft. by 66-in. Hardinge ball mill in closed circuit with a 12 by 32-ft. model FX Dorr classifier. These two mills operate as primaries and the classifiers overflow a product that is approximately 65 per cent minus 200 mesh.

The classifier overflow runs by gravity to two 6-cell No. 24 Denver Sub-A flotation machines which operate in parallel. The pulp enters cell 2 and passes through to cell 6, which overflows the primary tailings. The froth from cells 3, 4, 5 and 6 is returned to cell 1 for cleaning. The froth from cells 1 and 2 is thickened in a Genter thickener, then reground in a 4 by 20-ft. tube mill, which is operated in closed circuit with a 7-ft. bowl simplex Dorr classifier. This classifier overflows a product that is 95 per cent minus 325 mesh. The reground primary concentrate is then floated in an 8-ft. Southwestern air cell, producing the final concentrate and a cleaner tailing. The cleaner tailing is refloated in a 2-cell No. 24 Denver Sub-A machine, the concentrate from which is returned to the tube mill for further grinding, and the tailing is sent to the primary ball mills.

The primary tailing passes to two 10-cell No. 24 Denver Sub-A machines, which operate in parallel. These rougher machines produce a final tailing and a low-grade concentrate, which carries about 5 to 6 per cent sulfides and 80 per cent silica. The rougher concentrate is thickened in a 40-ft. Dorr thickener, then reground in an 8-ft. by 60-in. Hardinge ball mill operated in closed circuit with an 8 by 25-ft. model D Dorr classifier. The classifier overflow is 85 per cent minus 200 mesh, and it flows by gravity to a 4-cell No. 24 Denver machine for floating. The concentrate from this machine is returned to the primary ball mills and the tailing is sent to a 32-ft. Southwestern air cell. The Southwestern cell makes a low-grade concentrate, which joins the rougher concentrate, and a low tailing, which goes to waste. At the present time 1100 tons of ore is milled per day.

No. 1 *Grinding Unit* (See Fig. 4).—This mill regrinds 250 tons of rougher concentrate per day, and as the mill capacity is greater than is required for this work, it is also fed 130 tons of crude ore per day. The ball mill operates at 21.5 r.p.m. Manganese-steel liners are used and the ball load is maintained by the addition of 1½ and 2½-in. cast-iron balls.

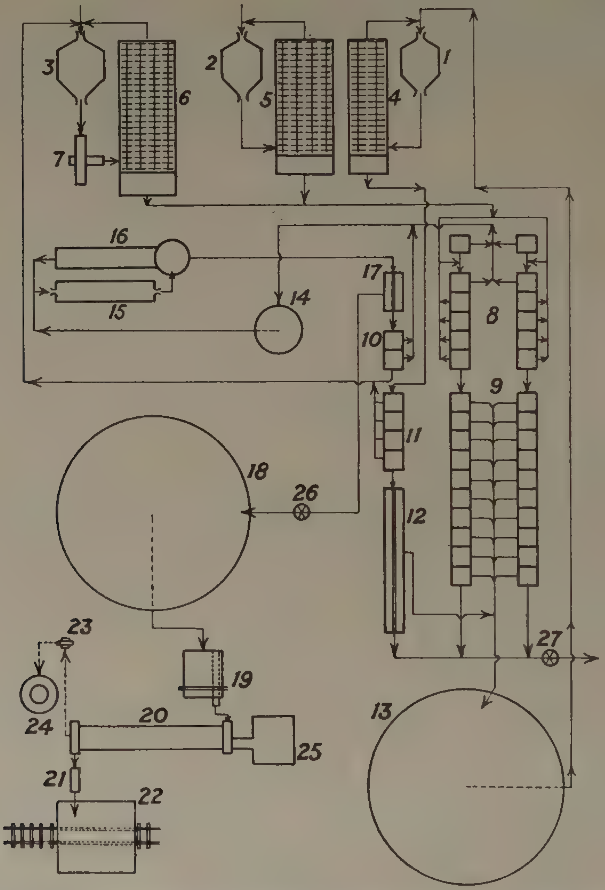

Fig. 4.—Concentrator flow sheet, Beattie Gold Mines Limited.

1. No. 1. ball mill
2. No. 2 ball mill
3. No. 3 ball mill
4. No. 1 classifier
5. No. 2 classifier
6. No. 3 classifier
7. Scoop elevator
8. Primary cells
9. Rougher cells
10. Two-cell cleaner
11. Middling cleaner
12. Air-cell cleaner
13. Middling thickener
14. Center thickener
15. Tube mill
16. Bowl classifier
17. Final cleaner
18. Concentrate thickener
19. Filter
20. Drier
21. Bucket elevator
22. Storage bin
23. Fan
24. Cyclone
25. Furnace
26. Concentrate sampler
27. Tailing sampler

Classifier speed, 6½ s.p.m.
Classifier slope, $1\frac{13}{16}$ in. per foot.
Classifier efficiency, 77 per cent.
Circulating load, 100 per cent.
Classifier overflow: Density 1.239 = 30 per cent solids. Alkalinity 0.009 per cent free Na_2CO_3.

No. 2 *Grinding Unit.*—This mill grinds 350 tons of ore per day. The ball mill operates at 21.5 r.p.m. Manganese-steel liners are used and the ball load is maintained by the addition of 2½-in. cast-steel balls.

Classifier speed, 20.5 s.p.m.
Classifier slope, 2⅞ in. per foot.
Classifier efficiency, 65 per cent.
Circulating load, 600 per cent.
Classifier overflow: Density 1.350 = 40 per cent solids. Alkalinity, 0.004 per cent free Na_2CO_3.

No. 3 Grinding Unit.—This mill grinds 625 tons of ore per day. The ball mill operated at 19 r.p.m. Manganese-steel liners are used and the ball load is maintained by the addition of 2½-in. forged-steel balls. This mill (10-ft. dia.) produces approximately 11 per cent more finished product per horsepower consumed than does the 8-ft. diameter mill.

Classifier speed, 15 s.p.m.
Classifier slope, $2\frac{5}{16}$ in. per foot.
Classifier efficiency, 65 per cent.
Circulating load, 1100 to 1200 per cent.
Classifier overflow: Density 1.350 = 40 per cent solids. Alkalinity 0.004 per cent free Na_2CO_3.

Attention is called to the fact that the 12-ft. FX classifier has the same efficiency when overflowing 625 tons per day (over 50 tons per foot) as has the 12-ft. D classifier when overflowing only 350 tons per day (30 tons per foot of width).

Tube-mill Grinding Unit.—This mill regrinds about 175 tons of primary concentrate per day. The mill operates at 31 r.p.m. White-iron pocket-type breast liners and manganese-steel end liners are used. The ball load is maintained by the addition of 1¼-in. cast-iron balls.

Classifier speed, 5 s.p.m.
Classifier slope, 1½ in. per foot.
Classifier overflow: Density 1.260 = 30 per cent solids. Alkalinity neutral.
Dry solids = 3.2 specific gravity.

Effect of Reagents in Grinding Circuits.—The addition of reagents to the grinding circuits has marked effects on the classifier efficiency. As the alkalinity of the primary circuits is increased up to 0.004 per cent free sodium carbonate, classification is benefited. Raising the alkalinity above this point retards classification and the classifier efficiency falls off rapidly. The effect of sodium carbonate in the primary concentrate regrind unit is to destroy classification completely, with the result that this circuit is kept neutral.[1]

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[1] See Classification. *Canadian Min. Jnl.* (July and August, 1934).

Reagents Used in Flotation.—The following reagents are used in the quantities indicated:

Added to grinding circuit, lb. per ton ore:	
Barrett No. 4 flotation oil	0.05
Soda ash	1.00
Copper sulfate	0.10
Added to primary flotation, lb. per ton ore:	
Sodium ethyl xanthate	0.07
Pine oil	0.05
Added to rougher flotation, lb. per ton ore:	
Rhodamine B solution	0.02
Pine oil	0.02

The Rhodamine B solution is a special reagent developed to float the low-grade middling product as a rougher concentrate. This concentrate has too low a mineral content to be activated by xanthate and the flotation accomplished is in the nature of a quartz float, with just sufficient sulfides present to give enough selectivity to separate the middlings from the tailings.

Dewatering of Concentrate

Thickening, filtering and drying are done in a separate building, to which the final concentrate is pumped at about 20 per cent solids. The concentrate is thickened in a 40 by 12-ft. Dorr thickener which, as it is considerably over capacity, acts as a storage tank whenever the filter is not in operation. The thickened concentrate goes to an 8 by 10-ft. Dorrco filter in which the moisture content is reduced to 12 per cent. The filter cake drops into a 54-in. by 30-ft. Ruggles-Coles single-shell drier, and the moisture is reduced to 5 per cent. The drier discharge is elevated by a bucket elevator to a 300-ton storage bin from which it is loaded by gravity into railroad trucks. To reduce the dusting loss during transportation, 5 per cent moisture is left in the concentrates.

Costs

The amount of ore milled per month is about 30,000 tons. The total costs for the month of April, 1934, averaged $0.8169 per ton of ore milled. These costs are fairly representative, and were distributed as shown in Table 7.

PROPOSED METALLURGICAL TREATMENT

Since the development of the Beattie orebody, a program of research work has been vigorously carried on in an effort to solve the problem of local treatment of the flotation concentrates. Sufficient progress has been made in the last six months to warrant the installation of a 200-ton cyanide plant. This research work now indicates that an over-all recovery of 75 per cent of the gold, as bullion, can be obtained by fine grinding, and pretreatment of the concentrates prior to cyanidation.

TABLE 7.—*Distribution of Costs*

	Per Ton Ore Milled
Direct costs	
Crushing	$0.0995
Grinding	0.4093
Flotation	0.1418
Dewatering concentrates	0.0291
Conveying	0.0272
Sand pumping	0.0096
Water supply	0.0092
	$0.7257
Indirect costs	
Supervision, research, etc	$0.0465
Assaying, lighting, heating, compensation and sundry charges	0.0447
	$0.0912
Total costs	$0.8169

Current tests indicate that by further pretreatment, a recovery of 95 per cent of the gold in the flotation concentrate will be made eventually. This will then represent a recovery of 82 to 84 per cent of the gold in the original ore.

At the present time a 2½-oz. concentrate is being shipped to a custom smelter. The tailing losses are high, owing to the necessity of producing a concentrate of this grade in order to cut down marketing costs per ton of ore milled. Marketing costs are approximately $1.10 per ton of ore treated, and this cost, together with high operating costs and excessive tailing losses due to the necessity for producing a high-grade concentrate, leaves a wide margin on which to work in favor of a local treatment plant.

The new flow sheet, which will be in operation in October, 1934, will be as follows:

First, a low-grade concentrate carrying about 1.2 oz. gold per ton will be produced in the present flotation mill. This low-grade material will be removed by the primary cells and sent to the cyanide plant (Fig. 5) before regrinding. The concentrate from the rougher circuit will be reground in the 4 by 20-ft. tube mill, which at present is employed to regrind the primary concentrate. The reground rougher concentrate will be returned to the primary ball mills. In this way, No. 1 ball mill will be released from regrinding service and will be available for primary grinding. This will have the effect of increasing the present concentrator capacity from 30,000 tons to 36,000 tons per month, with a consequent saving of about 10¢ per ton of ore in the concentrating costs.

The low-grade primary concentrate will be pumped to the cyanide plant, where it will be reground in a 6 by 16-ft. Vickers tube mill. The tube mill will operate in closed circuit with a Dorr bowl classifier having a 16-ft. bowl and a 12-ft. rake compartment. This classifier will overflow a product all minus 325 mesh. Grinding will be done in a lime circuit with no cyanide present.

The classifier overflow will pass to a 10 by 10-ft. Denver, Wallace-type agitator, and then to a 40-ft. Dorr thickener. The thickener overflow will go to waste. The underflow will be filtered and washed on a 10 by 16-ft. Oliver filter. The filter cake will be repulped with cyanide solution and agitated for 12 hr. in 16 by 18-ft. Dorr agitators, supplemented by the addition of a Denver agitator in each tank. The agitator discharge will go to a 40-ft. Dorr thickener. The overflow of the thickener will pass to clarification and precipitation, and the underflow will be given triple filtration on 10 by 16-ft. Oliver filters, the last of which will give the residue a clear water wash.

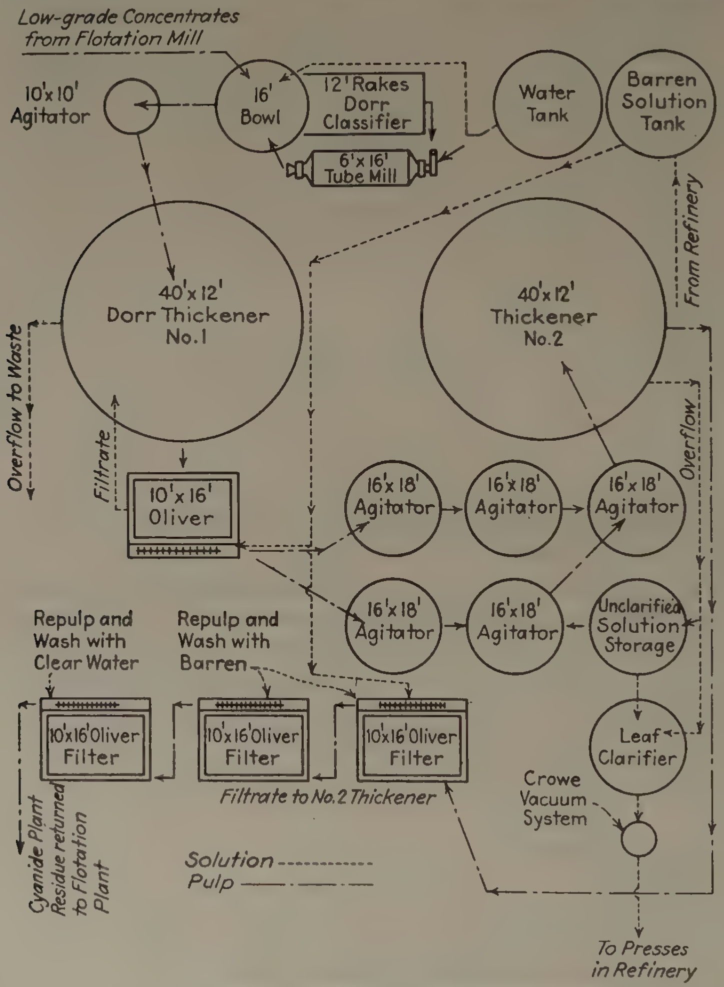

FIG. 5.—FLOW SHEET OF CYANIDE PLANT.

The cyanide residue will be returned to the concentrator, where it will be subjected to flotation treatment. The present final cleaner and regrind cleaner cells, which will be released from duty by the production of the low-grade primary concentrate, will be used for this purpose. For the time being, the concentrate produced by the flotation of the cyanide residue will be shipped to a smelter, but it is expected that when the new process is developed this material will be treated locally.

The advantages over the present method of marketing the concentrate are:

1. Concentrator tailing losses will be reduced about 0.015 oz. gold per ton; approximately $0.50 per ton, present price of gold at $35 an ounce.
2. Concentrator capacity will be increased 6000 tons per month without the installation of additional equipment, and a saving in operating costs of $0.10 per ton of ore will be effected by the increased tonnage.
3. About 75 per cent of the gold in the concentrate will be recovered as bullion. At the present time it is necessary to wait for three or four months for smelter returns.
4. An additional extraction of 15 per cent of the gold will be obtained, by refloating the cyanide residue, in the form of a low-grade concentrate containing about 0.70 oz. gold per ton, 38 per cent iron and 40 per cent sulfur. The freight and treatment charges on this material will be lower than on the present 2.5-oz. concentrate. When shipping the 2.5-oz. concentrate, the shrinkage amounts to about $0.15 per ton of ore milled. The same shrinkage when shipping a 0.70-oz. concentrate will amount to only $0.05 per ton of ore, which gives a saving of $0.10 per ton of ore milled on the item of shrinkage in shipping.
5. The cyanide plant will be in the middle of the process, and losses incidental to starting a new plant on a refractory ore will be recovered by the flotation of the cyanide residue.
6. There still exists in the Beattie metallurgy the problem of extracting the refractory gold remaining in the low-grade final concentrate mentioned above, without having to treat the latter by direct smelting. This problem is now being studied, and extractions obtained by certain treatments have been eminently satisfactory. At the time of writing, the biggest problem remaining is to evolve the technique of applying the newly discovered process to every-day operations. When this is done we will be in the pleasant position of having to treat a relatively small quantity of the daily tonnage to obtain the refractory gold.

ACKNOWLEDGMENTS

The authors wish to acknowledge the assistance of Mr. A. J. Keast, Manager of the Beattie Gold Mines, in the preparation of this paper, and to thank him and the Directors of the company for allowing the publication of details of the Beattie operation so soon after coming into production.

Milling Methods and Costs at Presidio Mine of The American Metal Co. of Texas

By Van Dyne Howbert,* Member A. I. M. E., and Fred E. Gray†

(El Paso Meeting, October, 1930)

The Presidio mine of The American Metal Co. of Texas is situated 45 miles south of Marfa, Texas, a town on the Southern Pacific R. R., and lies approximately 20 miles north of the Mexican border Communication with the railroad is maintained over a state highway. All supplies for mine and mill are hauled in by truck from Marfa and precipitates and concentrates are freighted to the railroad by the same means and shipped to Selby and El Paso, respectively.

The mill is at the town of Shafter, 1 mile from the mine and at approximately 350 ft. lower elevation. The ore is conveyed to it by an aerial tramway which requires practically no power on account of the difference in elevation.

The mill is a countercurrent decantation cyanide and gravity concentrating plant of 200 tons daily capacity. Coarse and fine crushing are done at the mill. Two near-by shallow wells in the bed of Cibolo Creek furnish an abundance of water. Power is generated for the mill and town by a Diesel engine plant.

Ore Treated

The ore is oxidized and siliceous. The greater part contains approximately 16 oz. of silver per ton, but mixed with this are many particles of ore of much higher grade, assaying in many instances 100 oz. or more. In the 16-oz. material there is an almost constant amount of silver which is difficult to recover by cyanidation. The high-grade material contains a much greater proportion of silver which is easily amenable to cyanide treatment. Thus, as the grade of ore becomes higher, the percentage of recovery of silver increases rapidly, under equal milling conditions.

The silver minerals contained in the ore are principally argentite and cerargyrite, the former predominating. The lead minerals, all of which are argentiferous, are chiefly cerussite and galena, with occasionally a little anglesite. The gold is free; most of the ore contains merely a trace

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TABLE 1.—*Average Mill Assays*

Product	Dry Tonnage	Ag			Au, Oz.	Pb			Zn, Per Cent.	Fe, Per Cent.	CaO, Per Cent.	MgO, Per Cent.	Mn, Per Cent.	Insol., Per Cent.	Moisture, Per Cent.
		Total, Oz.	Sulfide, Oz.	Chloride, Oz.		Total, Per Cent.	Sulfide, Per Cent.	Nonsulfide, Per Cent.							
Year 1929[a]															
Heads	54,644	19.741	11.901	7.840	0.02	2.499	1.253	1.246	4.87	3.87	12.84	6.15	0.38	48.9	3.0
Tails	53,575	1.916	1.916	Tr.	Tr.	1.640	0.790	0.850	4.26	2.81				50.0	
Precipitates	53.2	12666.64			14.57	38.840									
Concentrates	1,010.7	295.92			0.52	43.360									
One Week's Operation of Table Plant															
Table heads from Hummer screen		18.90				3.50	1.65	1.85							
Table heads from tube mill discharge		5.80				0.97	0.49	0.48							
Table concentrates		298.90				43.40	16.10	27.30	7.7	5.4				11.8	10.0
Table tails (combined)		3.50				0.70	0.49	0.21							

[a] 974,049 oz. Ag, 1,279 oz. Au, and 936,004 lb. Pb recovered.

but occasional concentrations run as high as 0.5 oz. per ton. Table 1 gives the analysis of average mill heads.

The principal constituent of the gangue is quartz. The ore is not particularly hard to break, though it is rather abrasive. Table 2 gives a screen analysis of the ore as delivered from the mine, where it has been previously passed through grizzlies spaced at 7 in. The ore is exceptionally dry, containing an average of approximately 3 per cent. moisture.

The tonnage of ore delivered from the mine is figured by weighing daily the buckets for two complete turns of the aerial tramway and using the average bucket content thus determined with the total number of buckets delivered for the day, as obtained from an automatic counter. The moisture content varies so slightly that moisture samples are taken only occasionally. The mill tonnage is calculated each day by subtracting or adding the difference in the amount of ore in the bin each morning, as determined by taking measurements at six specified points and thus arriving at the cubic contents of ore therein. The error of these daily measurements is adjusted at the end of a month by taking the total num-

TABLE 2.—*Screen Sizes*

Size	Run of Mine (Breaking Feed), Per Cent.	Intermediate Crushing Feed, Per Cent.	Hummer Screen Feed, Per Cent.	Hummer Screen Undersize, Per Cent.	Tube Mill Discharge, Per Cent.	Classifier Overflow, Per Cent.	Table Concentrates, Per Cent.	Table Tails, Per Cent.	Cyanide Plant Tails, Per Cent.
Inch ring									
+3	41.7	1.2							
−3 +2	11.9	2.0							
−2 Inch square +1	14.3	35.2							
−1 +⅝	5.4	15.5							
−⅝ +¼	10.6	24.3	20.4						
−¼	16.1	21.8							
−¼ Mesh +8	....	....	38.6	....	2.1				
−8 +20	....	....	18.1	23.3	1.4	....	7.6	3.2	
−20 +40	....	....	8.8	27.6	8.3	....	15.1	12.2	
−40 +60	....	....	3.8	9.4	12.2	....	11.7	11.6	
−60 +80	....	....	1.7	8.4	17.2	....	15.4	16.6	
−80 +100	....	....	0.8	3.3	11.6	2.9	9.5	10.9	2.6
−100 +200	....	....	2.8	11.3	21.1	27.5	28.4	14.6	27.4
−200	....	....	5.0	16.7	26.1	69.6	12.3	30.9	70.0

ber of tons of ore delivered to the bin for the month, and subtracting or adding the difference in the amount of ore in the bin from the amount there at the first of the preceding month, thus arriving at the monthly tonnage.

The average tonnage treated during the term of the present company's operations has been 157 tons per 24 hr. The grinding equipment could handle up to 300 tons per day but the agitation and settling capacity is not sufficient for more than 175 to 200 tons, depending on the grade of the ore.

HISTORY OF MILL

In the year 1883 the Presidio Mining Co. erected the first mill on the property, a small pan-amalgamation plant with a capacity of 50 tons per day. It was operated until 1913, when a cyanide mill was built on the same site, using the same stamps and part of the other equipment. When the present company, a subsidiary of The American Metal Co., Ltd., took over the property in April, 1926, the mill was substantially the same as in 1913. After a development campaign in the mine the company resumed milling operations in February, 1927. Since that time, the old coarse-crushing plant, which was at the mine, has been replaced by a new one at the mill, and the old stamp battery has been replaced by a new Symons cone crusher. A Hum-mer screen has been installed, the Wilfley table plant remodeled to better the lead recovery, and many minor changes have been made in various departments. The recoveries of silver and lead have been materially increased and costs reduced.

Table 3 gives a comparison of grades, recoveries and costs during past years. The costs prior to 1927 are approximate, as accurate data on operations of the old company are not available. Costs do not include coarse crushing. Tables 4 and 5 give the costs of operation for the year 1929.

TABLE 3.—*Grades, Recoveries and Costs*

Period	Total Tons	Mill Heads, Oz. of Silver	Recovery of Silver, Per Cent.	Direct Mill Cost per Ton
1883 to 1912 incl.	450,000	25.84	81.68	$4.73
1913 to 1926 incl.	720,000	12.00	83.66	2.55
1927	48,190	22.87	91.41	1.92
1928	57,475	23.17	91.04	2.07
1929	54,644	19.74	90.29	1.86[a]

[a] Does not include fine crushing. New crushing plant in operation in 1929.

Since the property has been operated by the present company, the average tonnage of ore milled per month has been approximately 4700,

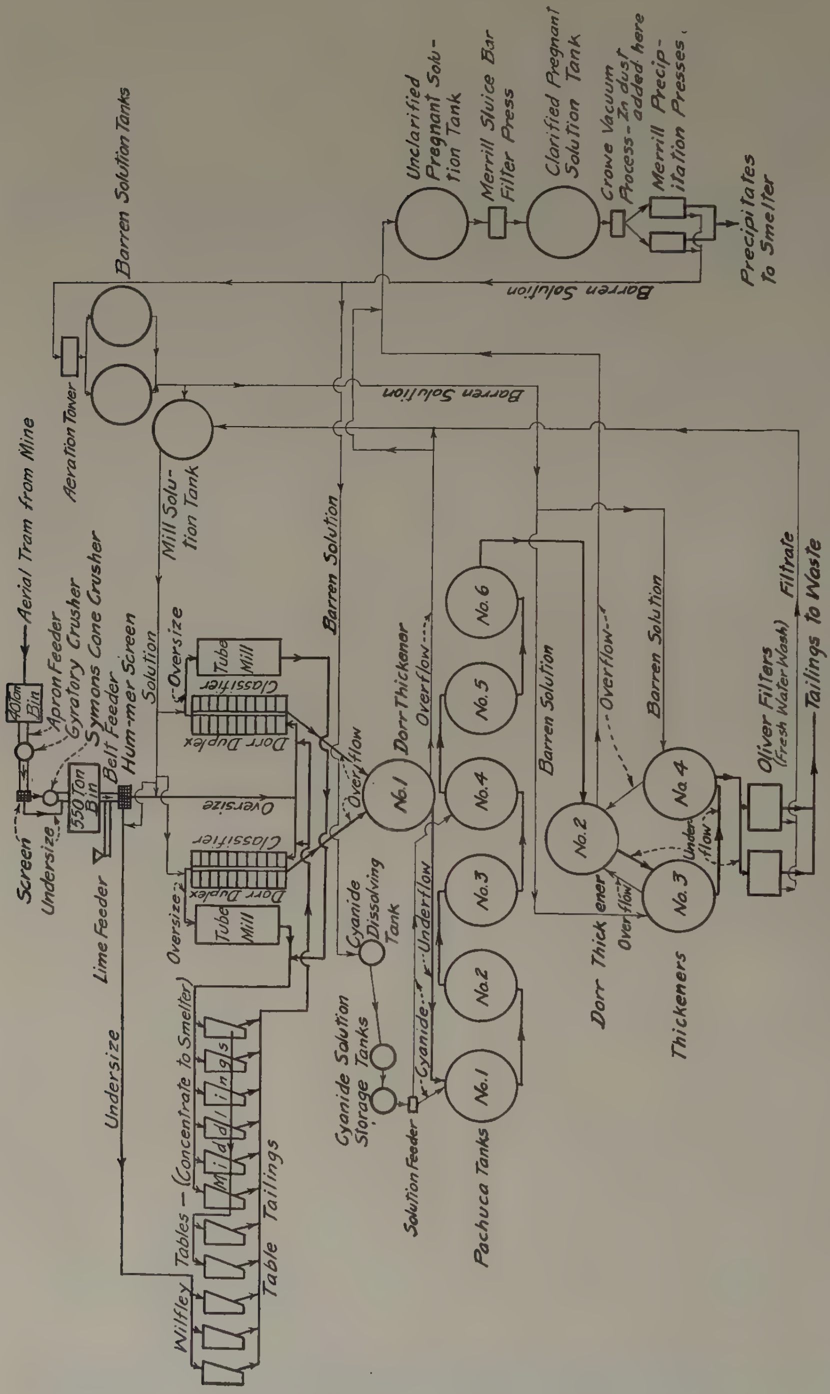

FIG. 1.—FLOW SHEET OF MILL AT PRESIDIO MINE.

carrying, in addition to the silver, $0.20 to $.050 per ton in gold and 2.0 to 2.5 per cent. lead.

It should be remembered that the mill was originally built for pan amalgamation and that in 1913, when it was changed to cyanidation, much of the same equipment was used, so that the present plant was not laid out to the best advantage as regards elevation and position of some of the machinery. During operation by the present company, a good many changes have been made and new equipment has been installed; the existing arrangement, of course, has greatly influenced such installations, and excuses must be made for what might otherwise appear to be

Table 4.—*Summary of Direct Costs*

Period covered, year of 1929. Tons of ore treated, 54,644.

	Operation		Repairs		Power[a]	Total
	Labor	Supplies	Labor	Supplies		
Breaking	$0.018	$0.002	$0.007	$0.006	$0.011	$0.044
Intermediate crushing	0.017	0.005	0.010	0.044	0.020	0.096
Total crushing	$0.035	$0.007	$0.017	$0.050	$0.031	$0.140
Tube milling	$0.067	$0.094	$0.025	$0.097	$0.148	$0.431
Agitation	0.057	0.005	0.011	0.021	0.082	0.176
Filtration	0.022	0.002	0.014	0.032	0.026	0.096
Precipitation	0.018	0.097	0.007	0.018	0.030	0.170
Drying and melting	0.024	0.018	0.001	0.001		0.044
Pumping and elevation	0.002	0.012	0.004	0.022	0.042	0.082
Tabling	0.083	0.018	0.014	0.022	0.018	0.155
Experimental	0.004	0.007				0.011
Chemicals	0.010	0.495	0.002			0.507
Supervision	0.186					0.186
Total milling[b]	$0.473	$0.748	$0.078	$0.213	$0.346	$1.858

[a] Cost per hp-hr. $0.01183.

[b] Not including cost of crushing.

Table 5.—*Summary of Costs, in Units of Labor, Power and Supplies*

Period covered, year of 1929. Tons of ore treated, 54,644.

	Operation	Repairs	Total
Crusher labor, tons per man per 8-hr. shift	85.92	227.70	62.38
Mill labor, tons per man per 8-hr. shift	7.05	48.43	6.16
Crusher power, hp-hr. per ton ore			2.62
Mill power, hp-hr. per ton ore			29.25
Lime, 70 per cent. available CaO, lb. per ton ore			10.78
100 per cent. NaCN, lb. per ton ore			2.37
Zinc dust, oz. per oz. Ag in precipitates			0.84

poor designing. The flow sheet of the mill as it is now being operated is shown in Fig. 1. Average mill recoveries are given in Table 6 and metallurgical data in Table 7.

POWER PLANT

The mill power plant consists of three De La Vergne Diesel engines, of same size and type, with a combined rating of 450 hp.; they are of the high-compression type. These engines drive an 8-in. line shaft, from which are driven three 115-volt d.c. generators delivering a total of 115 kw., two air compressors delivering 525 cu ft. of air per minute at 15 lb. pressure, and part of the mill equipment. The latter includes gyratory crusher, tube mills, filters, several pumps, etc. Power costs for 1929 averaged $0.01183 per horsepower-hour.

BREAKING

The tramway from the mine delivers the ore into a 40-ton bin ahead of the crushing plant. This bin is lined with old tube mill liners, which give very good service. From the bin, the ore is fed to the primary breaker on an 18 by 60-in. Allis Chalmers apron conveyor of the ratchet-feed type, driven by a 2-hp. motor. The amount of material delivered by the feeder is governed by a rack and pinion gate.

The primary crusher is an 8-A Telsmith gyratory, driven from the main line shaft of the mill by a system of countershafts and belts; sufficient electrical power is not available to drive it by motor. This machine receives feed with a maximum size of 7 in. and is set to break to a size between 1¾ to 2¼ in.; on this basis it has a capacity of approximately 35 tons per hour. Size of feed is given in Table 2. No provision is made for protection against tramp iron ahead of this crusher, other than the care exercised by the ore sorters at the mine and the tramway operators, but very little trouble has been experienced.

TABLE 6.—*Average Mill Recoveries*

Period Covered, Year of 1929

Product	Weight, Per Cent.	Recovery,[a] Per Cent.		
		Ag	Au	Pb
Heads	100.00	100.00	100.00	100.00
Tails	98.05	9.7	3.0	65.7
Precipitates	0.10	62.6	57.5	1.5
Concentrates	1.85	27.7	39.5	32.8
Precipitates plus concentrates	1.95	90.3	97.0	34.3

[a] Recoveries include corrections based on smelter returns.

TABLE 7.—*Metallurgical Data*

Period Covered, Year of 1929

Total tons ore treated	54,644
Days operated	365
Hours operated per day	24
Average tonnage per 24 hr	150
Total tons precipitates	53.2
Average tons precipitates per 24 hr	0.146
Total tons concentrates	1,010.7
Average tons concentrates per 24 hr	2.77
Recovery of silver, per cent	90.29
Recovery of lead, per cent	34.27
Recovery of gold, per cent	97.00
Ratio of concentration	54.06:1
Net water consumption per ton ore (mill and power plant), gal	960
Ball consumption per ton ore, lb	2.76
Liner consumption per ton ore, lb	0.68

INTERMEDIATE CRUSHING

The primary breaker product is discharged on to a 14-in. rubber conveyor belt, inclined at 22° from the horizontal and traveling at a rate of 186 ft. per minute. A Dings 15 by 15-in., 115-volt magnetic pulley acts as the drive pulley for the conveyor belt and protects the intermediate crusher from tramp iron. The conveyor belt discharges into a chute, fitted in the bottom with a ⅝ by ½-in. screen inclined at 50° from the horizontal, which removes the fines; although stationary, it rarely blinds because the ore is exceptionally dry.

The intermediate crusher is a 3-ft. Symons cone fitted with a fine bowl. It is driven by a 60-hp. motor connected by a 12-in. endless rubber belt. The machine is set to crush to ¼ in. and has a capacity at this setting of approximately 25 tons per hour. It consumes an average of 32 hp., with a peak of 50 hp. Screen analysis of the feed is given in Table 2.

The discharge joins the fines from the screen on a 14-in. rubber conveyor belt, inclined at an angle of 22° from the horizontal and traveling at a rate of 246 ft. per minute, which delivers it into the 550-ton storage bin. This is constructed of steel and has a natural ground flat bottom. The ore is fed from it at the bottom in the middle of one side, making about 300 tons available without shoveling.

SCREENING AND TABLING

The ore is taken from the mill bin on a 16-in. rubber conveyor belt, inclined at 25° from the horizontal, the amount being governed by a small rack and pinion gate. This belt travels at 60 ft. per minute and delivers to a 3 by 5-ft. Hum-mer screen, fitted with a plain chute feeder and a 10-mesh screen. It is vibrated by an 0.45-kw., 15-clyce, 115-volt a.c. generator, and is set at a 33° screening angle. With the average feed, containing over 30 per cent. minus-screen material, it maintains an efficiency of about 70 per cent. Screen analyses of feed and undersize are given in Table 2.

The fines from it, which contain an important proportion of the total lead in the mill heads, are tabled on three No. 5 Wilfley tables, which make lead-silver concentrate. The tailings from the three tables join with the screen oversize and enter a Dorr classifier, operating in closed circuit with a tube mill and eight No. 5 Wilfley tables. These eight tables make lead-silver concentrate from the tube mill discharge, and are protected from oversize by a stationary screen, through which the table feed must pass.

Grinding

The plant is equipped with two 4½ by 18-ft. El Paso Foundry tub mills, so arranged that they can be operated separately or together They are driven by 16-in. rubber belts from the main line shaft. The pinion shafts are equipped with 30-in. friction clutches. Cast steel balls are used, 90 per cent. 2½ in. and 10 per cent. 3 in. A ball load of approximately 20,000 lb. is carried, and consumption amounts to less than 3 lb. per ton of ore. A test of 40,000 lb. of chrome steel balls against the cast steel balls was made; in the absence of much impact, due to the small mill diameter, the cast steel balls proved much more economical. Cast steel liners, made in El Paso, are used. Liner consumption amounts to 0.68 lb. per ton ore, which is a material decrease from that obtained before the installation of the Symons cone crusher.

The tube mills turn at 28 r.p.m. Sufficient solution is used to maintain an average of approximately 76 per cent. solids in the tube mill discharge. The average circulating load is about 370 tons per 24 hr. A screen analysis of the tube mill effluent is given in Table 2.

Classification

The two model C-20 duplex Dorr drag classifiers, each 24 in. by 21 ft. 4 in., which may be run either jointly or separately as desired, travel at a speed of 20 strokes per minute. They are set at a slope of 2½ in. per foot. Classifier overflow contains an average of approximately 70 per cent. −200-mesh material, and is elevated by a 3-in. Wilfley sand pump to a 30 by 10-ft. tray Dorr thickener. The overflow contains an average of 17 per cent. solids. A screen analysis of it is given in Table 2.

AGITATION AND DECANTATION

The classifier overflow is thickened in No. 1 tray thickener, referred to above, to 40 per cent. solids. It is then pumped by a duplex Dorrco pump to the No. 1 Pachuca tank, which is connected in series to five other similar tanks. Four of the Pachucas are 14 by 28-ft. conical-bottom tanks, and two of them are 12 by 36-ft. conical-bottom tanks- Air pressure is maintained at 15 lb. per sq. in. and is furnished by a 12¼ by 12-in. Ingersoll Rand NE1 compressor, which has a capacity of 300 cu. ft. of free air per minute.

The last Pachuca tank in the series discharges into No. 2 Dorr thickener, which is also a 30 by 10-ft. tank. The thickened pulp, at 45 per cent. solids, is pumped by a duplex Dorrco pump into a sump, from which it is elevated by a 2-in. Wilfley sand pump to tank 3, which is a 30 by 10-ft. thickener of El Paso Foundry design. The underflow from it is discharged by gravity to tank 4, which is of same design and size as No. 3. The thickened pulp from No. 4 at about 50 per cent. solids is discharged by gravity to an 11½ by 14-ft. Oliver filter. An average of 315 tons per day of barren solution is added into thickeners 3 and 4 for decantation purposes. The overflow from them is returned to thickener 2, the overflow of which is discharged into an unclarified pregnant solution tank of 50 tons capacity. A part of the overflow from No. 1 tank is also discharged into the pregnant solution tank, the remainder being returned to the head of the mill. An approximate average assay of overflow solutions from each thickener shows Ag per ton solution as follows: No. 1, 3.0 oz.; No. 2, 4.5 oz.; No. 3, 3.0 oz.; No. 4, 1.5 oz.

PRECIPITATION

From the unclarified pregnant solution tank, the solution is pumped by a 2-in. Worthington OS volute pump through a 22-frame, 3½ by 3½-ft. Merrill sluice-bar clarifying press. The filter medium is 10-oz. cotton duck, which has an average life of about two months.

The effluent solution from the clarifying press is discharged into a 50-ton tank for clarified pregnant solution, from which it is drawn by vacuum into the Crowe-Merrill zinc-dust precipitation plant. This is equipped with a Gardner-Rix 6 by 4½-in. vacuum pump, a Worthington 5 by 8-in. triplex pump, a Merrill zinc-dust feeder and two 22-frame, 36-in. Merrill Type C precipitation presses. After the zinc dust is added, the solution passes through 160 ft. of 3-in. pipe before reaching the presses. The precipitation capacity is 900 tons of solution per 24 hr., but an average of only 575 tons is precipitated daily.

The filter medium consists of 8-oz. burlap placed against the leaves, covered with a sheet of unbleached muslin over which is put No. 230 filter paper. Formerly 10-oz. cotton duck was used in place of the burlap and was covered with filter paper. Some difficulty was encountered, however, with the canvas; within a few days its interstices would become filled with lime and the pressure in the presses would rise prematurely; also, cleaning of the canvas proved expensive. The burlap gives good service for about 30 days; then it is replaced and burned and the ash is added to the precipitates. The muslin is easily washed and lasts about one month, when it is replaced and burned. It has been found expedient to use filter paper on top of the muslin; it is inexpensive and simplifies washing of the muslin and forms a more impervious medium.

The press filtrate averages 0.04 oz. silver per ton of solution, and is lifted by a 2-in. Cameron centrifugal pump to an aeration tower of cascade type, from which it is discharged into two 3600-gal. steel storage tanks, for distribution by gravity to the head of the mill and to thickeners 3 and 4 for decantation purposes.

The precipitate presses are cleaned about every seven days. The precipitates are dried on a pan heated by an oil fire and are then screened to remove pieces of filter paper. This paper is burned and the ash is added to the precipitates. The precipitates are then placed in paper bags inside of canvas sacks, sealed and boxed. Two sacks, weighing approximately 125 lb. each, are packed in a box and shipped by express to Selby, Calif. Experimental work and experience have shown that under the conditions obtaining here it is more economical to ship the silver in precipitates than to melt and ship it as bullion, unless the grade of the precipitates should be below 8000 oz. silver per ton. The average grade of precipitates obtained in the plant has been about 11,800 oz., and last year averaged over 12,000 oz. per ton.

TAILINGS DISPOSAL

There are two Oliver filters of same size and design, either of which removes the solution from the underflow of the last thickener. Each is equipped with three fresh-water sprays, and the filtrate is elevated back to the head of the mill by a Deming 4 by 6-in. triplex pump. Approximately 12,000 gal. of water per day is used in washing by the sprays. The vacuum is maintained by a 10 by 12-in. Marsh vacuum pump, which is aided when necessary by a 10 by 10-in. Ingersoll Rand NE1 vacuum pump or compressor. The latter can be used also for additional compressed air for the Pachuca tanks, when needed.

The filter blankets are placed in contact with the filter screens, no burlap being used between them; practice has shown that better results are obtained by leaving off the burlap, although somewhat shorter blanket life results. The average efficient blanket life is approximately two months.

The filtered tailings are discharged into a launder and carried to waste in Cibolo Creek by the addition of the cooling water from the power engines. Dissolved silver loss averages approximately 0.07 oz. silver per ton of dry tailings.

Details of Gravity Concentration

The purpose of tabling primarily is for lead recovery and saving of cyanide, but incidentally it also aids in silver recovery. As mentioned before, two different materials are treated separately: the undersize from the Hum-mer screen and the material screened from the tube mill discharge.

The three Wilfley tables used for the Hum-mer screen undersize handle an average of about 32 tons in 24 hr. The percentage of solids in the feed is maintained at about 14. The tables run at a speed of 252 strokes per minute, with a ¾-in. stroke. The maximum capacity of these tables on this type of feed is about 15 tons each per 24 hr. The purpose of their operation is to recover as much as possible of the lead before it enters the tube mills, where it would be badly slimed. As about 50 per cent. of the total lead that is tabled is contained in the feed to these three tables, they play an important part in lead recovery. Their concentrates and tailings are not sampled separately, so that figures can be given only on the gravity concentration plant as a whole.

The eight tables for treating the tube mill discharge are identical with the other three except that the length of stroke is about ⅝ in. They handle several hundred tons in 24 hr. A large proportion of the lead in this material is badly slimed, so that the percentage of recovery is not very high. The average recovery of lead for 1929 in the gravity concentration plant, based on total lead in the mill heads, was 32.8 per cent., while the recovery of lead from the material that was tabled was somewhat larger, since there was, especially before installation of the Hum-mer screen, some lead that was slimed and overflowed the classifiers, and thus never reached the tables. The tube mill discharge runs into a bucket elevator, which raises the product 20 ft., giving it sufficient elevation to flow to the table plant located outside the main mill building. The elevator discharges on to a stationary screen, ⅛ by ½ in., set at an angle of 50° from the horizontal. The oversize returns to the classifier while the undersize is split between six of these eight tables. A middling product is taken from these six tables, which is carried by a launder to a small bucket elevator and delivered to two cleaner tables.

The tube-mill discharge pulp, which contains 76 per cent. solids, is diluted to about 46 per cent. for tabling by using a part of the classifier overflow as diluent. Better results would be obtained if the table feed could be diluted more, but existing conditions do not allow more solution to be introduced into the circulating load between the tube mill, tables and classifier. Barren solution is used for head water on the tables.

No equipment is being used for handling the table concentrates other than boxes at each pair of tables for catching them. These boxes are cleaned each day by shovel and the concentrates are spread on a concrete platform in the sun to dry. After about 8 hr. they are shoveled on to another concrete platform where they are sacked, about 140 lb. to the bag. The sacks are then sampled, tied, weighed, hauled by truck to Marfa and shipped by freight to the El Paso smelter. The concentrate shipments average 10 per cent. moisture.

The screen analyses in Table 8 give an idea of operating conditions in the gravity concentration plant.

TABLE 8.—*Screen Analyses, Gravity Concentration Plant*

	Table Feed from Hum-mer Screen			Table Feed from Tube Mill Discharge		
Mesh	Weight, Per Cent.	Ag per Ton, Ounces	Pb, Per Cent.	Weight, Per Cent.	Ag per Ton, Ounces	Pb, Per Cent.
+ 20	23.28	16.2		0.88	5.4	
+ 40	27.59	23.6		5.66	7.6	
+ 60	9.41	26.0		8.61	5.6	
+ 80	8.42	23.8		15.26	7.1	
+100	3.32	20.6		7.31	5.4	
+200	11.32	17.1		23.28	5.9	
−200	16.66	13.2		39.00	4.9	
Total....	100.00	18.9	3.5	100.00	5.8	0.97
	Combined Table Tailings			Combined Table Concentrates		
+ 20	3.24	4.9		7.60	347.6	
+ 40	12.22	6.0		15.05	417.2	
+ 60	11.55	4.0		11.74	409.8	
+ 80	16.59	3.8		15.44	307.2	
+100	10.86	2.8		9.48	214.6	
+200	14.59	3.2		28.38	202.0	
−200	30.95	2.5		12.31	230.2	
Total....	100.00	3.5	0.70	100.00	284.8	44.82

CHEMICALS

Lime is introduced by a feeder immediately ahead of the Hum-mer screen. The lime is purchased from El Paso and contains an average of 70 to 75 per cent. available CaO. It is rather coarse and, in order that it may be fed uniformly, it is put through grizzly bars, spaced 1½ in. apart, into a small bin of one ton capacity, from which it is fed on to a 10-in. rubber conveyor belt that discharges into the mill circuit. The feeder belt is equipped with a ratchet drive, which allows the amount of lime to be regulated, and further regulation is effected by a gate in the bin over the belt. Average speed of the belt is about 10 ft. per hour. On account of the rather high percentage of zinc in the ore, together with the requirements of zinc-dust precipitation, accurate control of the lime is necessary. Average consumption is about 10 lb. per ton of ore.

Aero brand cyanide is introduced at the first and fourth Pachuca tanks. It is first dissolved in a small tank to a solution strength of 10 per cent. by weight; the solution is agitated by air for about 4 hr. before it is used, in order that soluble sulfides may be removed thoroughly, and runs by gravity from the dissolving tank to two wooden storage tanks. One of these is connected to a cyanide feeder of the bucket elevator type of which the solution level is the same as that of the storage tank, thus allowing the feeder to operate 5 or 6 hr. without attention. The discharge from the feeder ordinarily is divided evenly between the two Pachucas, but can be changed as desired. Formerly, the cyanide was all added at the first Pachuca tank, but experience has shown that cyanide consumption is lowered by adding it as is done now, thus keeping a more uniform cyanide strength throughout the Pachucas.

A free cyanide strength of from 2.7 to 3.0 lb. per ton of solution is maintained in the Pachuca tanks. With this strength in the agitators, the lowest cyanide concentration in the plant is from 2.2 to 2.7 lb. in the first thickener. The aim is to keep the free cyanide as nearly as possible to 3.0 lb. per ton in the No. 4 Pachuca. If only 2.8 lb. is carried here, trouble arises when something happens to cause a sudden drop in cyanide strength, as it would drop to 2.4 or 2.5 before it could be stopped, which would result in higher tailings; but when a concentration of 3.0 lb. is carried, the drop would not carry it much below the "critical point" of about 2.8 lb. and no bad effects would result. Cyanide consumption is little higher at 3.0 lb. than at 2.8, so there is nothing in particular to be gained by using the lower strength, and at times there is much to lose.

The strength of free lime carried in the solutions has proved to be of greatest importance. In 1927, when the present company resumed milling operations after a shutdown for a development campaign in the mine, free lime was carried at from 0.5 to 1.5 lb. per ton of solution, in accordance with previous practice. Low cyanide consumption resulted for the remainder of the year, but in January, 1928, during a rather cold spell, the consumption jumped from 2 to over 4 lb. It was found that the solutions were badly fouled with zinc, the total cyanide having risen to the surprising figure of over 12 lb. per ton of solution. Bleeding of solutions was started immediately and experimental work was commenced in the laboratory to find a way to keep the solutions in good working condition. This work showed that by increasing the alkalinity to approximately 2.5 lb. of free lime per ton of solution, the double cyanides of zinc could be broken up, the zinc precipitated and the solutions thus freed of the greater part of the zinc, with a consequent regeneration of a large part of the cyanide. This was then put into practice in the mill, with the same results. The regeneration was slower and not as complete in cold weather as in warm.

Another difficulty then arose as a result of the higher alkalinity; namely, a decrease in grade of precipitates. Apparently there is a point of alkinity at which cyanide regeneration takes place properly and, at the same time, the silver precipitation by zinc dust acts as it should. If the lime changes from that "critical point" to any appreciable extent, trouble appears in one or the other of the reactions. Also, it has been found that this alkalinity point varies somewhat from time to time. By keeping a careful watch on precipitation results, and by keeping the lime as high as possible, without injuring precipitation, usually the solutions can be kept in fair condition. By careful work the solutions are now kept at about 2.9 lb. free cyanide and 2.4 lb. free lime per ton of solution; thus the precipitation filtrate contains about 4.75 to 6.0 lb. total cyanide per ton, which apparently is about as good as can be done by the present practice.

Another factor that plays an important part in both grade of precipitates and condition of solutions is the amount of zinc dust used. Until about 1929, the precipitation filtrate was tested with sodium sulfide to determine the amount of zinc necessary, on the assumption that as long as this reagent caused a dark precipitate more zinc should be added to cause complete precipitation of the silver. The fact that most of the silver would be precipitated before the lead would begin to precipitate was not realized. The press filtrate is now tested with nitric acid, which gives a sufficiently good test for silver without interference from the lead. The zinc is cut down until traces of silver remain in the filtrate, thus leaving some lead in solution. This not only reduces the zinc consumption, but keeps the solutions more free from zinc and raises the grade of the precipitates materially. By using the least possible amount of zinc much of the bad effect of high alkalinity on precipitation is eradicated.

Table 9 shows consumption of various mill supplies during the past three years.

TABLE 9.—*Consumption of Supplies*

Materials	Year		
	1927	1928	1929
Grinding balls, lb. per ton of ore	3.107	2.978	2.761
Lime (70 per cent. available CaO), lb. per ton of ore	8.858	8.806	10.785
100 per cent. NaCN, lb. per ton of ore	2.009	2.808	2.373
100 per cent. NaCN, oz. per oz. silver in precipitates	1.770	2.425	2.777
Zinc dust, oz. per oz. silver in precipitates	0.933	0.939	0.845

DETAILS OF CONVEYING

Table 4 shows that conveying costs, given as "pumping and elevation," are approximately $0.08 per ton of ore milled. That cost, however, does not include conveying in the crushing plant, nor does it include cost of launder linings. It is the cost incurred in operating the various pumps and bucket elevators used to raise the pulp from one part of the mill to another. Costs of operation of such machines as the Dorrco pumps at the thickeners and the triplex pump in precipitation are charged directly to their respective departments. For the most part, cost of conveying as given in the table is that for operation of equipment that would not have been needed if the mill had been designed properly.

Elsewhere, information has been given as to the sizes and speeds of the conveyor belts in the crushing plant. They have been in operation since early in 1929 and have carried only about 75,000 tons of ore, so as yet there has been practically no expense other than that for power and lubrication. Both conveyors are equipped with Dodge roller-bearing troughing idlers, on which there have been no replacements.

The Hum-mer screen oversize is washed, by means of solution, in a launder to the classifiers. This launder has a fall of 2½ in. to the foot and receives much abrasion from the ore. It is lined with cast-steel troughed liners made in 2½-ft. sections. They are cast thicker at the bottom, where they receive most wear, and weigh 70 lb. each. Replacements can be made in a few minutes. Each liner has a life of about one year. The fines from the Hum-mer screen are carried to the Wilfley tables in a 4-in. standard pipe with a fall of 4 in. to the foot. As the installation was made less than a year ago, no information can be given as to its life.

Launders in the gravity concentration plant are constructed of wood without lining; the wear is slight and practically no replacements have been made in the past five years.

The bucket elevator that raises the tube mill discharge has a vertical height of 20 ft. It is driven by a 5-hp. motor. The head and boot pulleys are 30-in. dia. by 15-in. face. A 14-in., 8-ply elevator belt is used. The buckets are 14 by 9 by 6 in., cast iron, and are placed 15½ in. apart center to center. The belt speed is 275 ft. per minute. Life of these belts has averaged one year.

The table tailings, which range from 30 to 40 per cent. solids, and amount to several hundred tons of solids per 24 hr., are pumped through a 3-in. pipe to the classifiers, some 20 ft. higher and 215 ft. distant, by a 2-in. Wilfley sand pump. Runners and follower plates have an average life of three months. This pump runs at a speed of 1500 r.p.m.

The classifier overflow is pumped 25 ft. vertically and 60 ft. horizontally by a 3-in. Wilfley sand pump, running at 1320 r.p.m., through a 3-in. pipe line. As this pump is working on a material of which 70 per cent. will pass a 200-mesh screen, runners and follower plates wear from four to six months.

Due to the location of No. 3 thickener, it is necessary to elevate to it through 15 ft. the underflow from No. 2 thickener, by means of a 2-in. Wilfley sand pump. Wear on this pump is very slight.

Sampling

Until the first of 1930, all sampling in the mill was done by hand. Prior to operation of the new crushing plant at the beginning of 1929, intermediate crushing was done in solution and consequently the mill head sample had to be taken from material with a size of approximately 1¾ to 2 in. As mentioned formerly, the ore contains a fair proportion of its values in high-grade pieces, which naturally makes sampling difficult. Hence, before 1929, mill head samples checked poorly with actual results. Since the existence of the new crushing plant, intermediate crushing has been done dry and the mill head sample has been taken of ¼-in. material, with much better results. However, even during 1929 marked differences occurred. Early in 1930, an automatic head sampler was installed. It is operated by water, which unbalances two receptacles, one on each side of a fulcrum, and is set to cut across the stream from the feeder belt every 2½ min.; it cuts a sample of about 400 lb. per day, and is giving somewhat better results than were obtained formerly.

Tailing samples are taken by hand from the cake on the discharge side of the Oliver filter, at three places every 30 min. In many instances, nothing of value could be determined regarding grade of ore or percentage recovery of silver by use of the mill head assay. Hence it has been the practice to report both the assay value and the calculated value, based on production plus tailings, of silver in the mill heads. It is apparent that neither figure is absolutely correct, but in general the calculated head has been of more value to the operator than the assayed one. In 1929, there were variations between the monthly assayed and calculated head values as great as 1.5 oz. silver per ton, yet the annual average values varied only 0.17 oz. per ton; in three years' operation, the contents of the heads from calculation exceeded the contents from assay by 12,238 oz. silver, which is a variation of about 0.35 per cent.

The tailing sample is divided into two parts; one part is dried and assayed and the other is thoroughly washed, dried and assayed, in order to determine the dissolved silver loss at the filters. Table concentrates are weighed and grab-sampled daily; concentrate shipments are pipe-sampled for moisture content, and a grab from each sack is taken for a general sample. Samples of precipitate shipments are obtained as the bags are being filled; a measured amount is taken when the bag is one-third full, repeated when two-thirds full, and again when full. Various samples of solutions and pulps are taken by hand throughout the mill daily, such as overflow solution from each thickener, solids and solution from No. 6 Pachuca, press heads, barren solution and grinding efficiency sample.

WATER SUPPLY

Water from the wells at Cibolo Creek is furnished by two pumps for the mill and town and one small one for the mine. The two former are 6 by 8-in. and 6 by 10-in. Dean triplex pumps with a total capacity of 250 gal. per minute; an average of 200 gal. is required. Practically the only water used in the mill, except occasionally for washing out a tank, is that for wash on the Oliver filters and to carry the tailings to waste. The water added to the circuit on the filters almost equals what is lost with the tailings, and from evaporation and small mechanical losses. The cooling water from the engines furnishes most of that needed to dispose of the tailings. The total amount used per minute in the mill and power plant, exclusive of domestic requirements, averages 100 gal. Cost of pumping during 1929 was $0.02 per 1000 gallons.

EXPERIMENTAL WORK

Testing has been done at several experimental plants to determine whether flotation alone, or a combination of flotation ahead of cyanidation, could be used to advantage on this ore; results of such tests have always indicated a lower economic return than is obtained from the present scheme of cyanidation and gravity concentration. Experimental work, both at the mill and at testing laboratories, has also demonstrated that, although some additional lead and silver could be recovered from the mill cyanide tailings by flotation, the grade of the flotation concentrate would be too low to cover costs of truck haul, railroad freight and smelting.

Milling and Cyanidation at Pachuca

By R. R. Bryan,* and M. H. Kuryla† Members, A.I.M.E.

Developments in Cyanidation, 1907 to 1934

The Compañía de Real del Monte y Pachuca started a 10-ton pilot plant in March, 1906. The distinctive features of the first cyanide installations at Pachuca were: grinding in cyanide solution, all-sliming and filtration of ore in which silver constituted the major value.

Loreto started with a 300-ton "all-sliming" plant, using jaw crushers, grinding in stamps, Chilean mills and pebble mills in cyanide solution, concentrating with Wilfley tables and Johnson vanners, classification by cones and Dorr classifiers, intermittent thickening by decantation, mechanical agitation in flat tanks by stirrers and centrifugal pumps, filtration in Butters filters and precipitation by zinc shavings. The "San Francisco" mill No. 1 was contemporaneous, using a rather similar flow sheet. We shall trace the main developments in the three large mills of the district to their present practice.

Developments in Large Mills of District

Crushing and Grinding.—The trend of development in this part of the work has been the same as throughout the milling world; viz., an increase in the degree of comminution of the ore in dry-crushing machines, the advance from pebble mill to rock mill and then to ball mill and the elimination of the stamp battery through the encroachment upon its field on the one side by finer dry crushing and on the other by more efficient wet grinding.

The single-stage crushing in jaw type crushers, used at the start, has been replaced by two stages; at first, both stages by gyratory type and later the present practice, which is the same in all three mills, consisting of gyratory as primary followed by cone as secondary and delivering a product that may be rated at ½ to 1 inch.

Primary wet grinding, at first performed by stamps and Chilean mills, is now by ball and/or rod mills; present practice differing widely in the three mills. Santa Gertrudis uses a 7 by 15 ft. rod mill, San Rafael

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uses one rod mill 5 by 10 ft. in parallel service with one ball mill 6 by 8 ft. Loreto uses large-diameter peripheral-discharge grate-mills in parallel service with center-discharge mills of smaller diameter, all using balls.

Secondary wet grinding has always employed the tube mill, but the grinding media have been successively pebbles, mine rock, iron or steel balls. Peripheral-discharge rock mills were used on this service at Loreto, which at one time practiced three-stage wet grinding. Present practice differs mostly in the character of the balls used, San Rafael employing a local, white-iron ball, Santa Gertrudis a local, cast-steel ball made in an electric furnace, and Loreto purchasing a tempered forged-steel ball.

Fig. 1.—Bird's-eye view of Loreto.

Classification.—Dorr classifiers were introduced at the start of cyanidation and are the exclusive means of classification today. Cones were continued in use for some years. Esperanza drag classifiers and Callow screens found application at Loreto for some time.

Thickening.—When the first cyanide mills started operations the charges were thickened by settling and decantation, a three to one pulp being the usual result. In 1908 the San Rafael mill started with the first Dorr thickeners and was soon followed by the other mills.

Concentration.—The early practice stressed gravity concentration to the point of recovering as much as 37 per cent of the silver and 33 per cent of the gold with a concentration ratio of 50 to 1. Santa Gertrudis mill, built in 1911, did not provide for concentration. San Rafael discontinued concentration in 1913, and Loreto in 1921.

Flotation has been given fairly thorough trials, but has not succeeded in equaling the economic results of cyanidation.

Agitation.—The first agitation was mechanical, using stirrers and centrifugal pumps to circulate the pulp through relatively low, large-diameter tanks, referred to as "flat tanks." The importance of more air was soon recognized and the Brown agitator, using an air-lift instead of a centrifugal pump, was introduced in 1907 at the San Francisco mill and gained rapid favor under the name of "Pachuca tank." At present, much of the compressed air is introduced through perforated rubber or canvas diaphragms.

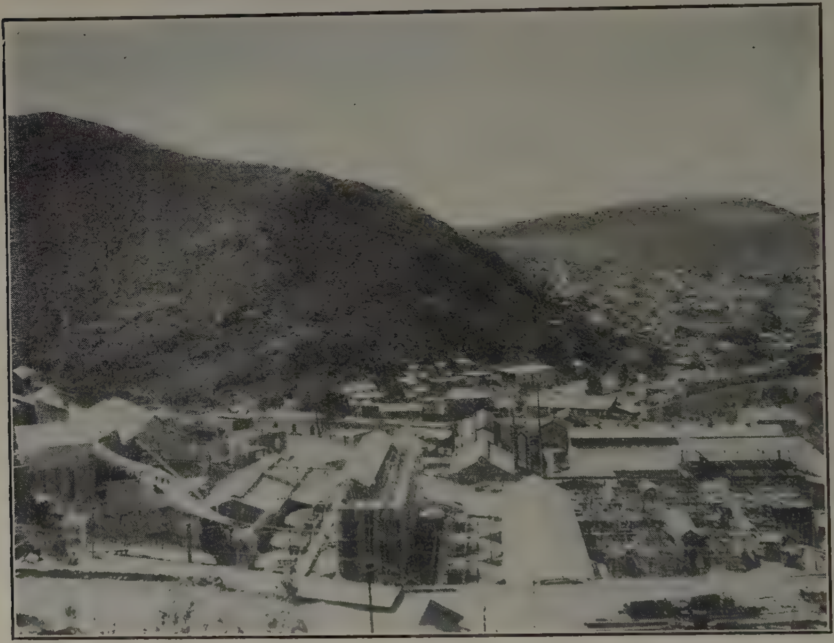

FIG. 2.—LORETO IN 1934.

Thin pulps, 3:1, were used at first. The Dorr thickener made a one to one pulp available, and that dilution is in use today. At first, the individual tanks were operated on charges or batches. The present continuous flow through a series of agitators was begun in 1911.

High lime, practically saturation, has always been carried and cyanide strengths have varied from 0.15 per cent to 0.35 per cent NaCN, depending on grade and character of ore.

Filtration.—Loreto originally installed Butters vacuum filters; San Rafael, the Moore vacuum filter and Santa Gertrudis, Merrill sluicing filter presses. Loreto and San Rafael have not changed their types of filters; Santa Gertrudis, when enlarging its capacity in 1920, installed

Butters filters ahead of the Merrill presses, thereby gaining a more efficient double filtration but at increased cost.

Clarification.—At the start, solutions were not clarified and precipitates were correspondingly low in grade. The first efforts to remedy this were by means of sand clarification, which progressed to present practice of either pressure or vacuum-type filtration.

Precipitation.—The Merrill zinc-dust process was a feature of the new Santa Gertrudis mill, started in 1911, and it rapidly replaced the original zinc-shavings precipitation in the other mills. The Crowe vacuum treatment was started in 1918. Zinc dust, emulsified in a small stream of barren solution, is pumped into the stream of pregnant solution coming from the main pumps. Loreto uses centrifugal pumps for both of these services while the other mills use plunger pumps.

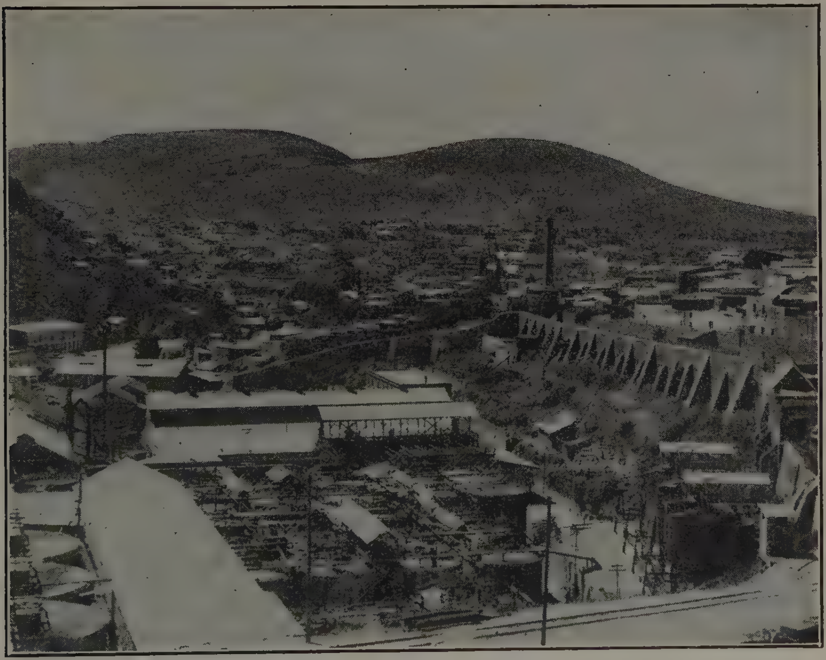

Fig. 3.—Loreto in 1934.

Melting Precipitate.—Melting was first done in graphite crucibles, then in tilting furnaces, and finally in oil-fired reverberatory furnaces. Both aluminum dust and electrolytic precipitation were tried and abandoned.

Cyanide Regeneration.—In 1924, cyanide regeneration was introduced at both the Santa Gertrudis mill and the Guerrero mill of Compañía de Real del Monte y Pachuca, accomplishing a considerable saving in the mechanical loss of cyanide.

Compared to the earlier processes, cyanidation has demonstrated the following advantages: lower operating costs, much higher gold recovery, and adaptiveness to a wider range of ores.

The district has a comparatively small tonnage of refractory manganese ores, upon which considerable pilot-plant work has been done using SO_2 and the Caron-Clevenger reduction. So far these special treatments have not proved economically justifiable.

Loreto Cyanide Mill, 1934 Operations

In 1906 work was started on converting Loreto from a 150-ton patio to a 300-ton cyanide mill. It has since been frequently remodeled and enlarged, and today it is the world's largest silver-cyaniding plant. The mill site is hemmed in by mountains and the city of Pachuca, but it is conveniently situated for ore delivery from various mines. Much ingenuity has been required to effect enlargements within the original walls and without interrupting operations.

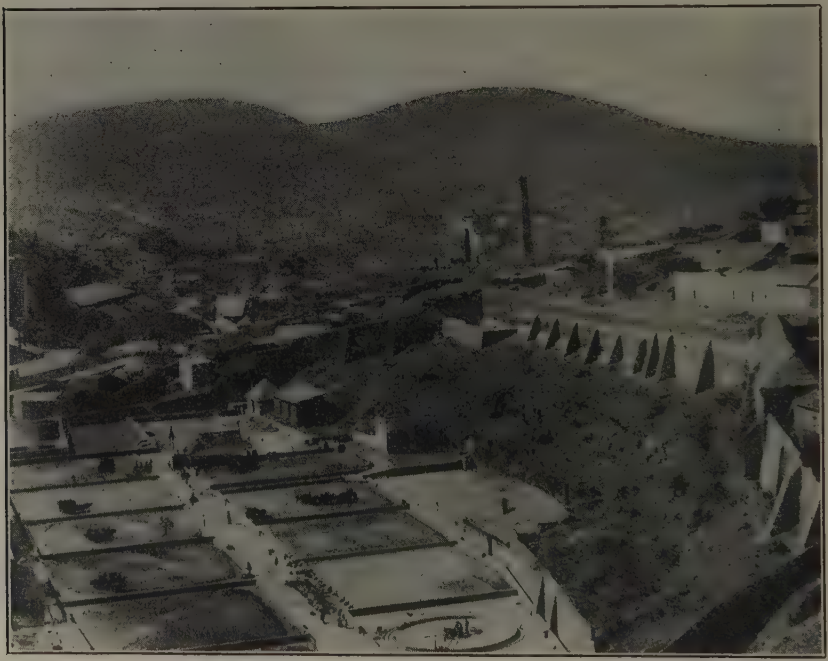

Fig. 4.—Loreto horse patio in 1891.

Crushing.—The crushing plant operates six days per week and 22 hr. per day, at an average rate of 200 tons per hour. The mine ore, maximum size 12 in., passes over a Miami grizzly, 40° slope, 4-in. clear openings. The oversize is crushed to a 4-in. product in a 20-in. Traylor gyratory crusher, belt-driven by 100-hp. motor using 28.5 hp. Cost of

TABLE 1.—*Grinding Data*

	Primary Grinding		Secondary Grinding	
Type mill	Grate	Trunnion	Trunnion	Trunnion
Make	Marcy	Traylor	Traylor	Local
Size, ft.	8 × 6	6 × 12	6 × 10	5 × 10
Number in operation	3	4	8	8
Horsepower per mill, installed	225	150	150	75
Horsepower per mill, used	246	160	134	77
Speed of mill, r.p.m.	23.2	23.1	23.1	28.0
Breast liners: material	Mn steel[a]	Mn steel[a]	Iron	Iron
Kg. per set	9650		10,000	7,300
Life, days	160	300	250	365
Grate bars, kg. per set	1560			
Chrome steel life, days	180			
Feed end liners: material	Mn St	Iron	Iron	Iron
Kg. per set	2259	1360	1360	820
Life, days	190	120	250	350
Classifiers: size, ft.	6 × 22	6 × 22	8 × 22	6 × 22
Strokes per minute	27.2	27.2	22.6	20.3
Per cent solids, overflow	64	64	20	20
Balls: size new, in.	5	5	2¾	2¾
Consumed per ton feed, kg.	0.662	0.647	0.732	
Original feed, tons per day per mill	660	450	310	165

SCREEN ANALYSIS, PER CENT

Mesh		Original Feed to Primaries	Product of Primaries, or Feed to Secondaries	Final Product of Secondaries
	On			
Inch	1.050	7.36		
	0.742	23.50		
	0.525	24.03		
	0.371	15.38		
Mesh	3	7.68	0.43	
	4	6.21	1.36	
	6	3.44	2.38	
	8	2.36	4.10	
	14	2.92	19.37	
	28	1.97	19.61	
	35	0.29	6.46	
	48	0.69	6.82	2.20
	65	0.53	4.41	7.90
	100	0.55	5.46	9.17
	150	0.55	4.73	13.13
	200	0.29	3.01	8.09
Through 200		2.25	21.86	59.51

[a] Scrap manganese-steel liners weigh 36 per cent of original weight.

supplies, including liners and mantles, is ₱0.011. (Costs, unless otherwise stated, are on a basis of total metric tons of ore milled and not on tons through the particular machine.) The product from the primary crusher, combined with the undersize of the first grizzly, passes over another grizzly of 40° slope, 2-in. clear openings, oversize direct to secondary crushers, undersize to a Traylor vibrating screen, 4 ft. by 6 ft. 6 in. with 1¼ by 1¾-in. clear openings, the oversize going to secondary crushers and undersize to fine-ore storage. Secondary crushing is performed by two Symons cone crushers, 5½ ft., set to give a ⅞-in. product, each driven by a 225-hp. motor through V-belt drives using 172 hp. each. Mantles and bowl liners are of manganese steel. The mantles weigh 1850 kg. new and 700 kg. worn, and last 85 days. The bowl liners weigh 2200 kg. new and 800 kg. worn, and last 87 days. Supplies, not including liners and mantles, cost ₱0.036 per ton, the main items being repair parts, oil and zinc.

Weighing and Sampling.—After secondary crushing the entire ore stream comes together on a 42-in. belt conveyor, inclined 18°, speed 310 ft. per min., passing over a Merrick weightometer having a capacity of 300 tons per hour. This weightometer is checked both by usual chain method and by passing weighed tonnages of ore over it. The ore stream is sampled by a chain-bucket cutter; the sample is successively cut by another chain-bucket and by two Snyder samplers, passing two sets of rolls for size reduction. The final sample is reduced by riffles and sample grinders.

Grinding.—Two-stage grinding in cyanide solution is practiced and two types of primary and secondary mills are in use. The principal data for both primary and secondary grinding will be found in Table 1.

Tempered steel balls are now used in both primary and secondary grinding. The first efforts to use tempered balls in 1932 were immediately successful with the small balls used in the secondaries, but the large balls used in primaries required a longer time to work out a successful technique. There is still considerable breakage of the large tempered balls, but these are now worked up by forging into small balls in our own shop at low cost. The consumption of steel balls before tempering and the present consumption of tempered balls is given in Table 2.

TABLE 2.—*Consumption of Balls*

	Consumption of Balls, Kg. per Ton	
	Untempered	Tempered
Primary mills	0.816	0.656
Secondary mills	1.240	0.732
Total ball consumption	2.056	1.388

Pulp Elevation.—In the remodeling and enlargements of 1929, local conditions made it impossible to obtain a gravity flow from primary to secondary milling and these departments had to be located on the same level and some distance apart. The pulp from the primary classifiers is pumped by one 8-in. Wilfley pump, V-belt driven by a 75-hp. motor, through a 10-in. pipe 196 ft. long. The screen analysis of material handled is given Table 1 under Product of Primaries. The pump handles 3800 tons solids and 2600 tons solution, or total of 6400 tons, pulp per day. The specific gravity of pulp is 1.55, so the pump is handling 757 gal. per min. The lift is 42 ft., which makes the static head 65 ft. of water, and the friction head is 23 ft., making a total head of 88 ft. The average continuous horsepower consumed is 72½ and the speed of pump is 810 r.p.m. All wearing parts are of white iron cast at Pachuca; runners last 7 days, follower plates 5.4 days and casings 30.5 days. Operating costs, exclusive of labor, amount to ₱ 0.012 per ton of ore.

Thickening and Agitation.—The pulp from the classifiers of the secondary grinding circuit is thickened to 48 per cent solids in nine thickeners, 48 ft. 9 in. by 15 ft. 3 in.

Two types of agitators are in use. The Pachuca, or Brown, agitator has been retained because of limited floor space, although showing higher operating costs than the flat agitators which have been used for all increases in agitation capacity. The flat agitator is a tank equipped with a Dorr thickener mechanism and air jets.

Total agitation capacity now consists of 20 Pachuca tanks, 15 ft. in diameter by 60 ft. high and 30 flat tanks, all of which are 30 ft. in diameter, 18 being 20 ft. high and 12 being 24 ft. high. The Pachucas are operated as two circuits and the flats as three. The compressors used to supply air for agitation have a total displacement of 13,550 cu. ft. per min. The Pachucas consume 290 cu. ft. free air per min. per tank at a pressure of 35 lb. per sq. in. and the flats consume 260 cu. ft. per min. per tank at 18 lb. The time of agitation is 73 hr. in the Pachucas and 70 hr. in the flats. The cost of air for agitation is ₱ 0.147 per ton of ore.

A study of the consumption of oxygen shows that 210 grams is consumed in agitation for the dissolution of 292 grams silver per ton of

TABLE 3.—*Free Air Supplied at Pachuca*

	Free Air Used per Dry Ton Slime, Cu. Ft.	Utilization of Oxygen, Per Cent
Pachuca agitation	7850	0.49
Flat-tank agitation	4070	0.96
Laboratory-bottle agitation	335	11.5

ore. This is 9.75 times the amount called for by Elsner's equation. To supply this amount of oxygen would require 38½ cu. ft. free air at Pachuca. The amount of air actually supplied per ton of ore in three different types of agitation is shown in Table 3.

To produce the same tail assay, the Pachucas require 73 hr., the flats 70 hr. and the laboratory bottle 36 hours.

About 35 per cent extraction is obtained in milling and thickening and 55 per cent in agitation, both referring to content of the original mill heads.

Aero brand cyanide, known locally as "surronide," is dissolved in barren solution to make a strong solution and this is added to the agitator heads in amount sufficient to bring them up to a strength of 0.25 per cent NaCN. Litharge is added to the cyanide in the dissolving tank to eliminate the soluble sulfides contained by the surronide. The addition of surronide amounts to 1 kg. equivalent NaCN per ton of ore and the cyanide recovered in the regeneration plant and returned to the mill solutions amounts to 0.62 kg., making the total consumption, without regeneration, 1.62 kilo NaCN per ton ore.

The consumption of lime is 6.0 kg. per ton ore. This lime is burned locally in vertical shaft furnaces, the product containing 70 per cent water-soluble alkalinity in terms of CaO. Each ton of lime produced requires 1.85 tons of limestone and 0.16 ton coke.

Filtration and Clarification.—Filtration is performed in six Butters vats containing a total of 1120 leaves each 67 by 117 in. giving 115,000 sq. ft. filtering area. Each vat averages 11 cycles per day of 128 min. each, leaving 32 min. per day lost time. The average cake is ⅞ in. thick and represents 0.308 ton dry slime per leaf. The cycle of 128 min. is divided into 20 min. for caking, 44 min. for barren wash, 5 min. for water wash to mill and 30 min. for water wash to regeneration, the remaining 29 min. being required for the various fillings, transfers and discharging. A vacuum of 18-in. mercury is maintained and each leaf passes about 0.01 ton solution or water wash per minute.

Each filter vat is operated by its individual wet vacuum pump. One dry vacuum unit was later installed for use on any one of the six vats, thereby avoiding filter delays while repairs are being made on wet vacuum pumps. This dry vacuum unit is superior to the wet system in faster caking, higher vacuum at all times and ease and simplicity of operation. The dry vacuum is supplied by one Ingersoll-Rand, type ER-1, 18 by 7-in. pump, operating at 350 r.p.m., piston displacement of 720 cu. ft. per min. and driven through V-belts by a 25-hp. motor. The pump is somewhat more than ample to operate one of the larger vats of 260 leaves and maintain a vacuum of 20 in. of mercury.

Filter leaves are cleaned by rotating brushes, and acid-treated every nine days in sulfurous acid of a strength equivalent to 12 kg. hydrochloric per ton, time in acid being 16 hr. Sulfurous acid was substituted for hydrochloric, eliminating the use of 0.26 kg. of 20° Bé. HCl per ton of ore. The sulfurous acid is made in a small tower in the regeneration plant through which the acid solution for the filter plant circulates and meets a great excess of SO_2 gas, the excess escaping to the acidification circuit of the regeneration plant. To prevent fouling, a constant amount of acid is bled from the acid-treating vats into the regeneration circuit and thus becomes effective in the acidification of the regeneration solutions. The consumption of sulfur is insignificant.

Clarification is performed in 11 Sweetland presses having a total area of 5080 sq. ft. filter leaf, the duty being 2.25 tons solution per day per square foot of filter area. Presses are discharged twice and cleaned once per 24 hr. and leaves are acid-treated every 10 days.

Precipitation.—Approximately three tons of solution is precipitated per ton of ore. Crowe vacuum system and Merrill presses are used. A departure from usual practice is the use of centrifugal pumps instead of plunger pumps for forcing the solution through the presses. The pumps used have been designed to give a relatively flat horsepower curve for their entire range of head capacities. Much pump and press trouble, due to the danger of the old system exceeding safe working pressures, has been eliminated. With the present system, pressure at the presses automatically regulates itself from 30 lb. minimum to 40 lb. maximum. In this range the pump efficiencies are 82 per cent.

The average consumption of zinc dust is 630 grams per kilogram of silver precipitated. Average analysis of the dried precipitate is:

	Per Cent		Per Cent
Silver	83.00[a]	Cu	0.10
Gold	0.46[b]	Insol	5.50
Fe	0.50	CaO	4.30
Pb	1.50	Se	0.25
Zn	2.20		
		Total	97.81

[a] 830 kg. per ton.

[b] 4.6 kg. per ton.

Moisture in undried precipitate is 32 per cent.

Cyanide Regeneration.—The mechanical loss of cyanide is reduced by means of cyanide regeneration. After the usual washes of the filter cake, including the permissible water wash to mill, the cake is given a further water wash of 30 min. and the values of cyanide and precious metals contained in this solution are removed in the regeneration plant, the cyanide being returned in a water-free form to the mill solutions; the silver and gold, as well as quantities of copper, are recovered in the form of a precipitate, which is shipped to the smelter.

The treatment of the solution is: (1) acidification with sulfur dioxide to neutralize lime and convert the cyanides to hydrocyanic acid, (2) vaporization of the hydrocyanic acid from this solution by means of a large current of air, (3) absorption of the hydrocyanic acid carried by this current of air in the regular mill solutions, (4) addition of zinc dust to precipitate the gold and (5) recovery of the precipitate of gold, silver and copper by means of filtration.

The regeneration plant treats 110,000 tons solution per month carrying 750 grams NaCN and 7 grams silver per ton. The tails assay 70 grams NaCN and a trace of silver. The consumption of sulfur is 2500 kilos per day and includes that used to make acid for the treatment of filter and clarifier leaves in the cyanide plant.

The cost per kilo of NaCN recovered is ₱ 0.22 without credit for leaf-treatment acid or for precious metals recovered.

Tailings Disposal.—Tailings leave the mill with 2.9 parts solution to one of ore and enter an 18-in. clay sewer-pipe line laid beneath the bed of the river, minimum grade 0.5 per cent. Emerging from the river at the lower end of the town of Pachuca, the tails are transported to one 8-in. Wilfley pump at the dam by means of an open ditch and then a flume 28 in. wide by 22 in. high, grade 0.35 per cent. Some solution is recovered from the dam by decantation and run through zinc boxes to recover the precious metals.

Having reached a height of 15 meters, the older parts of the dam have been abandoned for some time and dried out. During the windy season a great deal of dust arises from this part of the dam and considerable work has been done in an effort to abate this nuisance. The slopes have been planted with grasses and weeds and the top has been covered with a clay slime, classified from the tails. This slime dries, cracks and hardens into a suitable capping to reduce dusting.

TABLE 4.—*Loreto's Consumptions of Major Supplies, per Ton of Ore*

Cyanide purchased, in terms of kg. equivalent NaCN	1.00	Power, kw-hr., crushing and grinding	17.7
Grinding balls, kg	1.39	Cyanidation, air	5.3
Zinc dust, kg	0.26	Cyanidation, other	7.4
Lime, kgs	6.00	Miscellaneous	1.4
Sulfur, kg	0.66		
Labor, man-shifts (₱4.50 per 8 hr.)	0.11	Total	31.8

Recovery.—Recoveries in the district depend on economic considerations of metal prices, exchange rate, cost of cyanide and power, as well as on such refractory constituents of the ore as pyrolusite and excessive base sulfides. On "clean ores" the recovery reaches 93 per cent silver and 95 per cent gold. On heads of 400 grams silver and 2 grams gold, the present recovery in the district is about 90 per cent silver and 92 per cent gold.

Consumption of Supplies and Costs.—Loreto's consumption of major supplies, including labor and supplies, is given in Table 4.

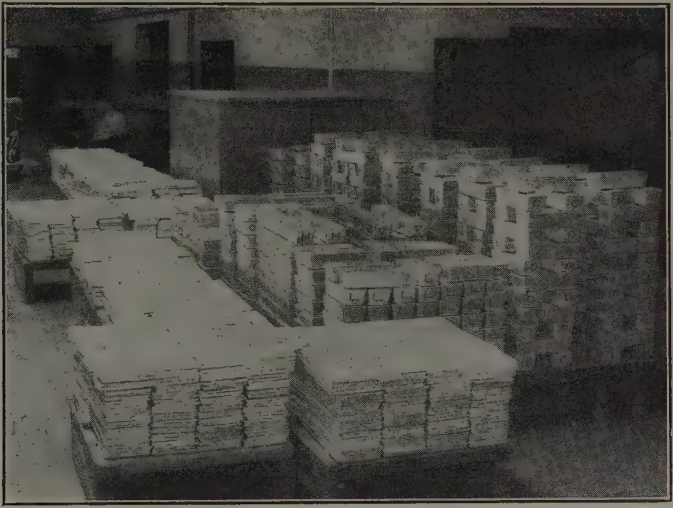

FIG. 5.—SILVER BARS AND ANODES IN REFINERY AT LORETO.
1,500,000 oz. in sight (about 20 days production at refinery).

Because of Loreto's disparity in capacity, it is thought more appropriate to cite present costs in one of the smaller cyanide mills of the district (Table 5).

TABLE 5.—*Costs at a Small Cyanide Mill in the Pachuca District*
400 TONS PER DAY

	Pesos per Ton		Distribution, Pesos per Ton
Crushing and grinding	1.27	Labor	1.38
Cyanidation	1.75	Supplies	2.04
Precipitation and melting	0.30	Power	0.72
Water supply	0.14	Miscellaneous	0.24
General expense	0.92		
Total	4.38	Total	4.38

The prevailing rate of exchange in 1934 was 3.60 Pesos per U.S. dollar.

Cyanide Milling Practice of The Fresnillo Company

By W. E. Crawford,* Member A.I.M.E.

The Fresnillo Company's mill at Fresnillo, State of Zacatecas, Mexico, treats oxide ores from the Fresnillo mine, dump ores and underground ores from the Plateros district, company leases and custom ores from the Zacatecas district, custom ores from other districts and tailings from old dumps at Fresnillo. The normal capacity of the mill is approximately 2600 dry short tons per 24 hours (a ton of 2000 lb.).

The final product of the mill is a precipitate containing gold and silver, which is smelted and shipped as doré bullion to the refinery of the Compañía Minera de Peñoles, S.A., at Monterrey, State of Nuevo Leon, Mexico.

The geology of the district was described in 1923, by T. C. Baker,[1] from whose paper the following data have been taken:

The mineralization of the district was effected in the Tertiary period, following the intrusions and preceding the appearance of the flows. The area within which productive veins have been found is 1 mile long by ½ mile wide. The important units of each of the four general fracture systems meet in the central portion of Proaño hill, which is an outcrop of graywacke, and where the rock was also intensely fractured by subsidiary movements, resulting in a shattered zone 1200 ft. long by 400 ft. wide.

The mineralization was more intense in the immediate vicinity of the principal channels through which the solutions rose. The valuable metals are silver and gold, the ratio by weight of the former to the latter being 1000 to 1.5. Argentite is the predominant silver mineral, and is associated with pyrite and manganese mineral. The sulfides are primary throughout. A slight secondary action is represented by small quantities of native silver and silver halides, principally cerargyrite, which are found in the upper part of the mine. Oxidation has penetrated with great thoroughness to the lowest horizon so far exposed, this being 420 ft. below the present ground-water level.

The shattered mass within the central portion of the hill and near the surface was sufficiently mineralized to produce a large stockwork

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[1] T. C. Baker: Fresnillo Glory-hole Mining Practice. *Eng. & Min. Jnl-Pr.* (1923) **116**, 931–942, 972–973.

deposit, which, on account of the conditions favorable to low mining costs, can today be profitably exploited.

Power and Water

Power is generated by the company in its own steam-turbine plant. The oxide mill requires 22.84 kw-hr. per ton of ore milled, and consumes 49.72 per cent of the power generated. Power is transmitted from the point of generation to the mill substation at 2200 volts. From the substation it is distributed at 2200 volts, 440 volts and 110 volts for use in the different departments.

Water for operating purposes is obtained from the lower levels of the operating mines, from shallow wells and from reservoirs that are fed by streams during the rainy seasons. The water is slightly alkaline and contains a high percentage of soluble salts.

Water is scarce during the dry season and is carefully conserved. The mill tailings are impounded for settling and the clear solution is returned to the mill circuit for further use.

Crushing

The ores are delivered from the mine bins and railway cars from outside districts to the crushing plant in 10-ton cars, which are weighed over track scales before entering the car tipple ahead of the crushing plant. The cars are dumped into a surge bin ahead of a 30-in. McCully crusher, driven by a 150-hp. induction motor. This machine is set at 3½ in. The product is conveyed to grizzlies with 1½-in. spacing above two Symons 48-in. horizontal disk crushers set at 1¼ in. The undersize from the grizzlies and the discharge from these two machines are then conveyed to Leahy 4 by 6-ft. heavy duty screens, where the minus ⅝-in. product is removed and from which the oversize goes to two 48-in. Symons vertical disk crushers, ordinarily set at ⅜ in. The products from the screen and the Symons vertical disks are then brought together and conveyed to the mill bin storage, where they are distributed over the length of the bin. This bin has a capacity of 6400 tons[2] of ore.

With the cessation of glory-hole mining, the character of the feed changed, and during the rainy season the operation of the crushing plant is complicated greatly by the adverse effect of the water on the clay in most of the ore. The ore becomes sticky and the capacity at times drops to less than one-third of normal. So far this problem has been met by opening the crushers and by attempting to keep them clean through the use of water and high-pressure air. Owing to the lack of headroom, sufficient screening and washing devices preliminary to crushing are not possible, without making expensive plant changes. The wet season ordinarily is short, but in some years it is very severe at times.

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[2] When tonnage is mentioned in this paper, the reference is to a short ton of 2000 pounds.

All of the conveyor belts are 30 in. wide, run 250 ft. per minute and have an inclination of 18°, except the one running from the gyratory crusher to the Symons horizontal disks, which is 36 in. wide.

Tramp iron is removed from conveyor 1 by a 42-in. suspended Ohio Magnet, and a magnetic head pulley on belt No. 2 is used to further protect the Symons vertical disks from accidental iron and steel.

The consumption of manganese steel in the crusher plant is 0.036 lb. per ton of ore crushed. The power consumed for crushing and conveying is 1.69 kw-hr. per ton of ore.

The crusher plant is equipped with a dust-collecting system to remove the dust created by the machines. The dust-laden air is drawn through an air washer before being liberated to the atmosphere. The solution and the sludge are drawn from the bottom of the washer and laundered to the slimes discharge of the sands-slimes separation plant.

From the mill's storage bin the ore is fed to six inclined conveyor belts, each 24 in. wide, through 8-in. pipe chutes in the bottom of the bin. Each belt feeds a primary fine-crushing mill. This primary grinding section consists of four 6-ft. 6-in. by 12-ft. 3-in. Marcy mills, grouped in two units, and one unit of two Hardinge ball mills; the secondary grinding consists of three 6 by 14-ft. Traylor ball mills. The products from each Marcy mill unit and from one Traylor mill are discharged into an 8 by 26-ft. Dorr duplex bowl classifier. The bowl overflow is discharged to the cyanide plant and the oversize is returned to the Traylor mill for regrinding. The discharges from the two Hardinge mills and a Traylor ball mill are treated in a like manner.

Each Marcy mill is driven by a 150-hp., 1170-r.p.m., slip-ring induction motor, operating through an Allis-Chalmers speed reducer to give 17 r.p.m. to the mill, and each Hardinge mill by a 200-hp., 390-r.p.m., slip-ring induction motor directly connected to the pinion shaft. The Hardinge mills are operated at 20 r.p.m. The Traylor mills are each directly connected to a 200-hp., 390-r.p.m., slip-ring induction motor and revolve at 24 r.p.m.

Each Marcy mill grinds 370 short tons per 24 hr.; each Hardinge mill has a capacity of 250 tons.

The Hardinge mills are loaded with 4¼-in. white iron balls; the Marcy rod mills with high-carbon steel rods 12 ft. long by 3-in. dia., and the Traylor mills with 3⅛-in. white-iron balls. The consumption of high-carbon steel rods in the Marcy mills is 1.66 lb. per ton of ore. The Hardinge mills use 2.65 lb. of 4¼-in. balls per ton of ore; the Traylor mills use 0.86 lb. of 3⅛-in. balls per ton of original feed.

The life of the liners in these mills depends upon the material from which they are made. The consumption of manganese-steel liners in the Marcy rod mills is 0.22 lb. per ton of ore ground and 0.126 lb. per original ton of feed in the Traylor ball mills. White-iron liners in the Marcy mills are consumed at the rate of 0.46 lb. per ton and 0.214 lb. in the Traylor mills. Chrome-molybdenum liners cast in an electric furnace in the company's foundry are being tried out, but at present no reliable data are available.

The power consumption for primary grinding is 7.47 kw-hr. per ton of ore ground and 4.44 kw-hr. for secondary grinding.

A fourth Traylor mill in closed circuit with an 8 by 26-ft. Dorr duplex classifier is used to grind the oversize classified out from the tailings that are pumped from the Santa Ana dumps.

The screen tests of the products of the various units are given in Table 1 and the final products to the sands-slimes plant in Table 2.

TABLE 1.—*Screen Tests of Products*

Mesh No.	Mesh Opening, In.	Marcy and Hardinge Feed		Marcy Discharge		Hardinge Discharge	
		Per Cent	Per Cent Cum.	Per Cent	Per Cent Cum.	Per Cent	Per Cent Cum.
	2.000	0.00	0.00				
	1.050	4.08	4.08				
	0.742	17.36	21.44				
	0.525	25.17	46.61				
	0.371	14.41	61.02				
+ 3	0.263	10.16	71.18				
+ 4	0.185	7.12	78.30			tr.	tr.
+ 6	0.131	3.65	81.95	tr.	tr.	0.50	0.50
+ 8	0.093	3.83	85.78	0.20	0.20	0.80	1.30
+ 10	0.065	3.01	88.79	1.40	1.60	1.70	3.00
+ 14	0.046	2.08	90.87	3.80	5.40	2.60	5.60
+ 20	0.0328	1.81	92.68	8.80	14.20	4.60	10.20
+ 28	0.0232	1.25	93.93	8.60	22.80	6.30	16.50
+ 35	0.0164	0.87	94.80	10.80	33.60	7.10	23.60
+ 48	0.0116	0.76	95.56	10.80	44.40	8.35	31.95
+ 65	0.0082	0.80	93.36	8.10	52.50	8.75	40.70
+100	0.0058	0.93	97.29	7.90	60.40	9.60	50.30
+150	0.0041	0.68	97.97	6.80	67.20	7.50	57.80
+200	0.0029	0.24	98.21	3.10	70.30	4.20	62.00
−200	0.0029	1.79		29.70		38.00	
Total........		100.00		100.00		100.00	

The grinding in each unit is made to conform with the grade of the feed in order to obtain maximum economic extraction. Considerable

TABLE 2.—*Final Products to Sands-slimes Plant*

Mesh No.	Mesh Opening, In.	Traylor Mill Product		Santa Ana Feed to Classifier		Final Product to Agitation Tanks		Final Product to Sand Tanks	
		Per Cent	Per Cent Cum.	Per Cent	Per Cent Cum.	Per Cent	Per Cent Cum.	Per Cent	Per Cent Cum.
	0.371			2.30	2.30				
+ 3	0.263			3.20	5.50				
+ 4	0.185			4.30	9.80				
+ 6	0.131			3.40	13.20				
+ 8	0.093			3.80	17.00				
+ 10	0.065			6.50	23.50				
+ 14	0.046			7.40	30.90				
+ 20	0.0328	tr.	tr.	6.10	37.00				
+ 28	0.0232	0.05	0.05	8.30	45.30				
+ 35	0.0164	0.50	0.55	8.00	53.30				
+ 48	0.0116	3.62	4.17	10.50	63.80				
+ 65	0.0082	10.13	14.30	9.90	73.70	8.00	8.00	60.00	60.00
+100	0.0058	13.75	28.05	8.20	81.90	11.00	19.00	25.00	85.00
+150	0.0041	11.50	39.55	3.50	85.40	12.00	31.00	7.00	92.00
+200	0.0029	7.00	46.55	3.00	88.40	11.00	42.00	5.50	97.50
−200	0.0029	53.45		11.60		58.00		2.50	
Total....		100.00		100.00		100.00		100.00	

variation in the fineness of the products results from treating ores of which the gross values range from $2.25 to $10.00 (U.S.A. currency) per ton. As a result of this practice ores from the Zacatecas district and some of the richer silver ores are ground fine enough so that the overflow from the bowl classifier is sent over a chip screen directly to the distribution box ahead of the Dorr thickeners.

The bowl-classifier overflow, containing the lower grades of silver ores and the reground Santa Ana tailings, is sent to two 6 by 26-ft. Dorr bowl duplex classifiers in the sands-slimes separation plant. The bowl overflow from the two classifiers is then passed over a chip screen and laundered to the distributor box ahead of the Dorr thickener, where it joins the high-grade gold and silver slimes. The sand discharge from the two classifiers is laundered to a sump ahead of the sand leaching plant, which will be described later.

The slime pulp is then distributed to six 10 by 60-ft. Dorr thickeners. The thickening mechanisms make one revolution every 10 minutes and are driven from two line shafts, each powered by a 10-hp. motor. The pulp is thickened to one part of solids to 1.75 parts of solution. The cone of each thickener is connected to a quadruplex Dorrco pump, which lifts the pulp to launders leading to a sump connected to the suction of two Krogh 5-in. sand pumps that elevate the pulp to Dorr agitators.

The Dorr agitators are divided into two continuous circuits, one containing six tanks, the other seven. The pulp is proportioned according to the number of tanks in each circuit. Each agitator is 25 ft. deep and 40 ft. in diameter. The agitators are equipped with Dorr thickener mechanisms, making a revolution every 3 min. and 40 sec. Agitation is accomplished by introducing compressed air under 22 lb. of pressure into the cone at the bottom of the tank. Owing to the clayey nature of the ore and the coarse grinding of the lower grade ores, the tanks require frequent cleaning to keep them up to capacity. This operation requires from 4 to 5 hr. per tank per month.

CYANIDATION

Cyanide is added at the head of the agitating circuit to build up the strength of the solution. The starting strength of the solution in the agitators is maintained at 0.125 per cent KCN. The lime is added as an emulsion throughout the circuit, so that the strength at the end of the treatment is 0.04 per cent. The consumption of lime is 16.2 lb. of 100 per cent CaO per ton of ore treated, and the consumption of Aero brand cyanide is 3.34 lb. per ton treated. The time of treatment in the tanks under normal operations is 53 hours.

The two end agitators are used as storage tanks for the Butters filter. The pulp from these tanks is delivered to four Butters filters through a 20-in. pipe line. The flow is controlled by hydraulic valves from a manifold in the center of the filter house. Each filter box has an individual 18 by 18-in. wet Gould vacuum pump, which maintains a vacuum of 18 to 19 in. of mercury during washing, as compared to a total possible vacuum of 22.5 in. at this altitude. Each filter has 156 standard leaves, 10 ft. 6 in. wide and 5 ft. 6 in. deep. The cycle on the Butters filter has a total duration of 100 min. Of this time 17 min. is devoted to caking of pulp, 34 min. to a barren solution wash, 24 min. to a water wash and 25 min. to the operation of filling and discharging. The water wash is made up of treated solution, from which the cyanide has been removed in the cyanide recovery plant, and enough water to maintain the plant balance of solution. In order to minimize the reducing action of the acid-treated solution from the recovery plant on the soluble silver in the cake, the solution is first made alkaline by the addition of lime emulsion and then aerated by pumping to and over a metal screen that is over the water-storage tank, in order to restore some of the oxygen content destroyed by the SO_2 treatment. After the cycle is completed the thick pulp discharged from the leaves is sent directly through launders to a pulp-storage sump. As the discharge thins down, the thin pulp is distributed between four Dorr dewatering tanks, 15 ft. deep and 40 ft. in diameter, where it is thickened. The discharge from the bottom of the tank is pumped by four Dorrco duplex diaphragm pumps to the pulp-storage sump. The combined pulp in this sump is then pumped by two 6-in. Wilfley sand pumps to the tailings dam. The pulp is discharged to the dam at a dilution of 1 to 1.

The filter leaves are treated every 10 days with dilute SO_2 solution, made by passing the gas from the roasting plant through water. The consumption of filter leaves amounts to 30 leaves per month on the four pulp filters and clarifier.

The leaching rate is maintained at 2 tons of solution per filter box on the barren solution wash. It is interesting to note that a higher leaching rate results in a cracked cake when the box is in the process of being emptied and filled between the different stages of the cycle. Cracks permit the short-circuiting of solution and poor washing then results.

The sand plant, during normal operations, handles 17 per cent of the total tonnage on the charge system. There are five 15 by 60-ft. steel tanks, each having a capacity of 1815 dry tons of sand. The sands delivered to the sump ahead of the tanks by a launder from the sands-slimes separation plant are elevated by a 5-in. Krogh sand pump to the tank charging into a diversion box connected to individual Butters distributors by a series of launders, arranged so that the sand can be diverted to a tank that is charging. The time of filling is anywhere from three to six days, dependent upon the amount of sand produced in the mill. When the tank is full it is allowed to drain and a strong bath containing 0.25 per cent KCN and 0.05 per cent CaO is added. This wash is normally continued for five days.

After the strong bath has been drained, a weak bath containing 0.075 per cent KCN and 0.05 per cent CaO is added for a period of five days.

Upon the termination of the weak bath the final water wash of 660 tons is added. After draining under a low vacuum the tank is discharged on to conveying belts through chutes over the conveyors; the remainder is shoveled into the chutes by contract labor. At the head of the conveyor belts the sand is mixed with the slimes discharged from the filter plant; the combined tailings flow by gravity to the tailings pond, where they are impounded. The sand has a moisture content of 14 per cent when discharged from the tanks. This low moisture helps to conserve water.

Part of the thickener overflow, and all of the solutions from the treatment of the ore, with the exception of the later stages of the weak washes and some of the barren washes, are clarified in a standard 156-leaf Butters filter. The low grade of the weak and barren washes and the remainder of the thickener overflow are returned to the milling circuit.

The clarified solution, amounting to 7000 tons daily, flows by gravity through a 12-in. pipe to the precipitating plant. Here the solution passes through the Merrill-Crowe vacuum process for removal of the dissolved oxygen before the zinc dust is added. This installation consists of three 6 by 12-ft. receivers, each connected to an Ingersoll-Rand 5 by 10-in. Imperial type No. 15 vacuum pump, driven by a 5-hp. motor. The de-aerated solution is then pumped by two 13 by 14-in. Aldrich triplex pumps to six 35-frame, 54-in. Merrill precipitating presses. The zinc dust is added as an emulsion at the discharge of the large triplex pumps by a small triplex pump. This system of introducing the zinc dust into the discharge of the pump has two advantages; (1) that of cutting out the wear on the valves and pistons of the larger pumps, and (2) that of reducing the possibilities of theft around the pumps.

The Merrill presses are cleaned every ten days and the recovered precipitate is smelted into doré bullion in a reverberatory furnace. The fluxing of the precipitate requires 5 per cent broken glass, 1.5 per cent of borax glass, 1 per cent manganese dioxide and 1 per cent of old iron. The bullion is cleaned, weighed and shipped to the refinery. The doré bars assay from 850 to 920 oz. in silver and 4 to 6 oz. in gold. The impurities consist of copper, lead and zinc.

Cyanide Recovery Plant

Under normal operating conditions the mechanical loss of cyanide in the filter operations is an important factor in cyanide consumption. This loss is due to imperfect washing and largely to the fact that the water wash is limited to the amount of water that is necessary to maintain a balance of plant solution.

This difficulty is practically eliminated by using barren solution, from which the cyanide has been removed, to extend the washing period. The removal of the cyanide is accomplished by treating the barren solution with SO_2 gas in the Merrill-Crowe recovery plant. The cyanide is liberated as hydrocyanic acid gas, which is absorbed in the alkaline mill solution in the absorption towers. It is interesting to note that the first commercial installation using the process was at Fresnillo and that much pioneering had to be done in order to overcome the early operating difficulties.

For details of the process the reader is referred to C. W. Lawr's excellent paper.[3] The plant that he describes is very similar to the installation at Fresnillo, the principal difference between the two plants being that the Fresnillo plant removes the cyanide from the barren solution, using SO_2 gas obtained by the roasting of pyrite concentrate from the Fresnillo Company's concentrator, whereas the plant of the Compañía de Santa Gertrudis, S.A., at Pachuca, removes it from an extra water wash after the plant balance has been satisfied, using SO_2 gas obtained by burning 98 per cent sulfur.

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[3] C. W. Lawr: Cyanide Regeneration or Recovery as Practiced by the Compañía Beneficiadora de Pachuca, S.A., Mexico. *Trans.* A.I.M.E. (1930) **87**, 488.

One point has been developed in the Fresnillo practice of using treated barren solution; namely, that it effects an attractive saving in cyanide, but, at the same time, the slightly acid solution has a precipitating effect on the silver in the filter cake—as noted under filter practice. Also the process introduces another adverse factor, because some SO_2 is absorbed with the HCN gas by the mill solution in the absorber towers and this SO_2 gas forms lime salts that absorb the oxygen in solution, which must be replaced by mechanical means in order to obtain the best metallurgical results, especially when treating gold ores. Wherever headroom is available, perforated screens are used under pump discharges to obtain an intimate contact between the solution and the air. Experiments are now being conducted with a 27-in. turbomixer in the mill-solution storage tank, to increase the oxygen content. A preliminary test of one hour raised the oxygen in 380 tons of mill solution from 68.5 to 89 per cent of saturation; in practice this one mixer raises the oxygen content of the mill solution 6 per cent.

The operation of the cyanide recovery plant is very simple and requires only one man per shift under the direction of the cyanide solution operator. During a normal month (for instance, July, 1934), 75,000 tons of solution was treated. The heads titrated 0.054 free cyanide, 0.073 per cent total KCN and 0.038 per cent CaO. The final treated solution titrates 0.006 per cent KCN.

To avoid frequent shutdowns of the main plant required to clean the first tank, the acidified solution is first passed through four tanks 4 ft. 4 in. in diameter and 10 ft. high. In these small tanks the solution comes into contact with a large number of wooden grids, which are arranged so that they can be easily hoisted with a chain block and cleaned. These grids collect the precipitate and by having a number of spare units they can be removed and cleaned at leisure, without any loss of operating time. This procedure has not only eliminated the shutdowns but has done away with the laborious and expensive operation of removing baffles from the large tank in the main circuit.

Santa Ana Tailings Reclamation Plant

The operation of this plant deserves mention because coarse sands and considerable gravel are pumped to the cyanide plant through a 6-in. pipe line 6540 ft. long against a static head of 147 ft. The material being handled represents the tailings from a lixiviation plant that treated old "patio" tailings.

At the time that attention was first given to these tailings it was estimated that one dump of 135,000 tons assaying 0.5 gram of gold and 150 grams of silver could be treated at a small profit, even though the price of silver at that time was $0.30 (U.S.A.cy.) an ounce, provided the tailings could be delivered to the mill at low cost. The transportation by railway was first considered, but the idea was rejected on account of the high cost of transportation and the inconvenience of transferring material from the cars to the mill. It was then decided to pump the tailings to the mill with a series of sand pumps. Several pump manufacturers were consulted, but the installation and contemplated operation were so unusual that very little information could be obtained. One manufacturer even went so far as to state that it was inadvisable to pump directly from one pump to the suction of another, and that sumps should be provided for the intake of each pump to eliminate entrained air.

The original installation was so arranged, and it was found impracticable to maintain the proper balances at the intake boxes between the incoming and outgoing pulp. The pipe line was then directly connected to the suction of each pump. The original installation consisted of two 5-in. sand pumps equipped with open impellers and one 6-in. pump that required a flooded suction. It quickly developed that the latter pump entrained enough air to shut off the flow after operating a short time. Vents were then placed in the line, but these proved unsatisfactory, because it was impracticable to build them sufficiently high to overcome the pressures that were developed at times in the line. The trouble was finally overcome by replacing the 6-in. pump with the other type of sand pump.

The installation now consisted of three 5-in. standard sand pumps, arranged in series, with each pump discharging into the suction of the following pump, respectively through 2580 ft., 2940 ft. and 1050 ft. of 6-in. pipe, the latter discharging into the mill classifier. A second 6-in. line conveys mill solution to be used in pulping the sands and slime.

In the early operations the wear on impellers, liners and pump shafting was so great that steady operation was extremely difficult. The engineering department, after studying the problem, redesigned the shell, liners and impellers and introduced gland water to protect the shafting at a higher pressure than the pressure in the pumps. These improvements—combined with regular, periodic renewals, resulted in more continuous operation.

The pulp dilution, L to S, is 4 to 1—equivalent to a specific gravity of 1.142. The velocity of the pulp in the pipe line is 6 ft. per second; the friction loss is 3 ft. of head per 100 ft. of 6-in. pipe.

When the operations were smoothed out, the record showed that the pressure on the pipe line at station 1 was 85 lb.; at station 2 it was 86 lb., and at station 3 it was 75 lb. When these three pumps were operating, any change in dilution or lag in power would necessitate the washing of the line with mill solution. As the product to be handled became sandier this trouble increased and tonnage dropped off. To remedy this defect a fourth pump was installed at Santa Ana in series with the first pump. This extra pump raised the pressure at Santa Ana pump station No. 1 to 125 lb. and at station 2 to 100 lb., but the pressure at station 3 remained at 75 lb. The fourth pump gives the added pressure that is needed to keep the sands moving in the pipe line, and no time has been lost since it was installed. Pumps 1, 3 and 4 are operated at 1190 r.p.m. by 75-hp. motors; pump 2 is driven at 1050 r.p.m. by a 50-hp. motor.

Shell liners and impellers are made out of white iron and the approximate life of a set is 250 hr. The pumps consume 8.65 kw-hr. per ton of dry tailings delivered at the mill. The total consumption of power for the total operation is 9 kw-hr. per ton.

The sand is shoveled into side-dumping cars, hauled by mules and dumped into a collecting bin, which feeds a 20-in. conveyor belt. This belt elevates the sand to a trommel, fitted with rail grizzlies, which scalps the larger rock, and an outside screen, which removes the plus ½-mesh rock remaining in the sand. This plus ½ product and the rock is fed into cars and trammed to the waste pile. The minus ½-mesh product is sluiced with mill solution to a Devereux agitating tank, and more mill solution is added until the proper dilution of 4 to 1 is obtained. Emulsified lime is added to this tank to destroy the acidity of the sands and slimes and to maintain the protective alkalinity of the cyanide solution. The pulp is then pumped to the mill for regrinding and cyanidation.

From 550 to 650 dry tons of sands and slimes are pumped daily. Screen tests show that an average of 9.8 per cent of the product remains on a 4-mesh screen and that only 18.1 per cent is minus 100-mesh material.

Higher silver prices and lower operating costs have made it possible to handle what originally was considered worthless material, and to date nearly 550,000 tons of tailings have been pumped to the mill. The 6-in. pipe line, carrying the pulp, has been turned a quarter of a turn twice to distribute the wear, which is greatest on the bottom sector of the pipe. The life of this line is remarkable, considering the character of the material carried and the fact that the pipe was not new when it was put into service.

Plant for Treatment of Manganese-silver Ore

It is a well-known fact that silver ores containing manganese dioxide are often difficult to treat with the cyanide process when the silver values are locked by the manganese. Manganese is found in varying percentages in the Fresnillo ore, and although, fortunately, the average tenor is not enough to necessitate special treatment, ores from some parts of the mine contain sufficient manganese to affect seriously the extraction of the silver. To improve the extraction on this relatively small quantity of refractory ore, S. P. McClusky, formerly metallurgist with the Fresnillo Company, devised a method of application of what has become known as the sulfur dioxide process for manganiferous silver ores.

Briefly, this method consists of: (1) grinding the ore in water; (2) subjecting the pulp to the action of sulfur dioxide gas in order to dissolve the manganese minerals; (3) precipitating the dissolved manganese with a lime emulsion; (4) aerating the pulp and finally, (5) cyaniding in the usual manner. The ideas involved in the method are: (1) that part of the silver is in too close association with the manganese minerals, which inhibit the action of cyanide solution on this silver; (2) that when these minerals are dissolved by SO_2 the associated silver is liberated and thereby becomes accessible to the solvent action of the cyanide solution. Moreover, if the dissolved manganese is then precipitated by lime emulsion and oxidized to the manganic state by aeration, it no longer adversely affects extraction, although it still remains in the pulp.

The tenor of the ore in silver, and the gain in the extraction by sulfur dioxide treatment, together with the cost of the process, are the criteria by means of which the applicability of the process to manganiferous silver ores may be judged.

The following description of the plant, with the accompanying flow sheets (Figs. 1 and 2), gives the main details of the process as applied at Fresnillo. The numbers on the flow sheets from 1 to 16 represent the direction of travel of the pulp; from 17 to 25, the flow of the SO_2 gas.

The ores requiring special treatment are collected in the general mill ore bin, so that they may be fed to either one of two Marcy rod mills (No. 1 of the flow sheets). This feed is ground in what is known locally as "spent" solution (No. 31 of the flow sheets)—a tailing solution from the cyanide recovery plant.

The "spent" solution contains only a small amount of cyanide, usually less than 0.008 per cent KCN, and is slightly acid on account of SO_2 gas. After grinding, the pulp is diluted with additional "spent" solution to a ratio of 1.5 solution to 1 of ore.

The fineness of the grinding averages 30 to 35 per cent passing through a 200-mesh sieve, and about 1 per cent coarser than 10 mesh. The pulp then flows to a 4-in. Wilfley centrifugal pump (No. 2), which delivers it to the top of the first and second of the sulfur dioxide treatment towers, which are sealed, airtight chambers, three in number (Nos. 3, 4 and 5 respectively). These towers are of wooden construction 3 by 3 ft. in cross-section and 17 ft. high, with wooden baffles lined with white-iron plates, arranged as shown on the annexed vertical section of a tower on Fig. 2. The purpose of the baffle plates is to disperse the pulp as it falls through the tower, so that it may come into intimate contact with the ascending current of SO_2 gas, the flow of which will be described later. The stream of pulp is about equally proportioned between the first and second towers (Nos. 3 and 4). The portion entering the second tower (No. 4) exits at the bottom and flows by gravity to the first conditioner tank (No. 6). The portion entering the first tower (No. 3) exits at the

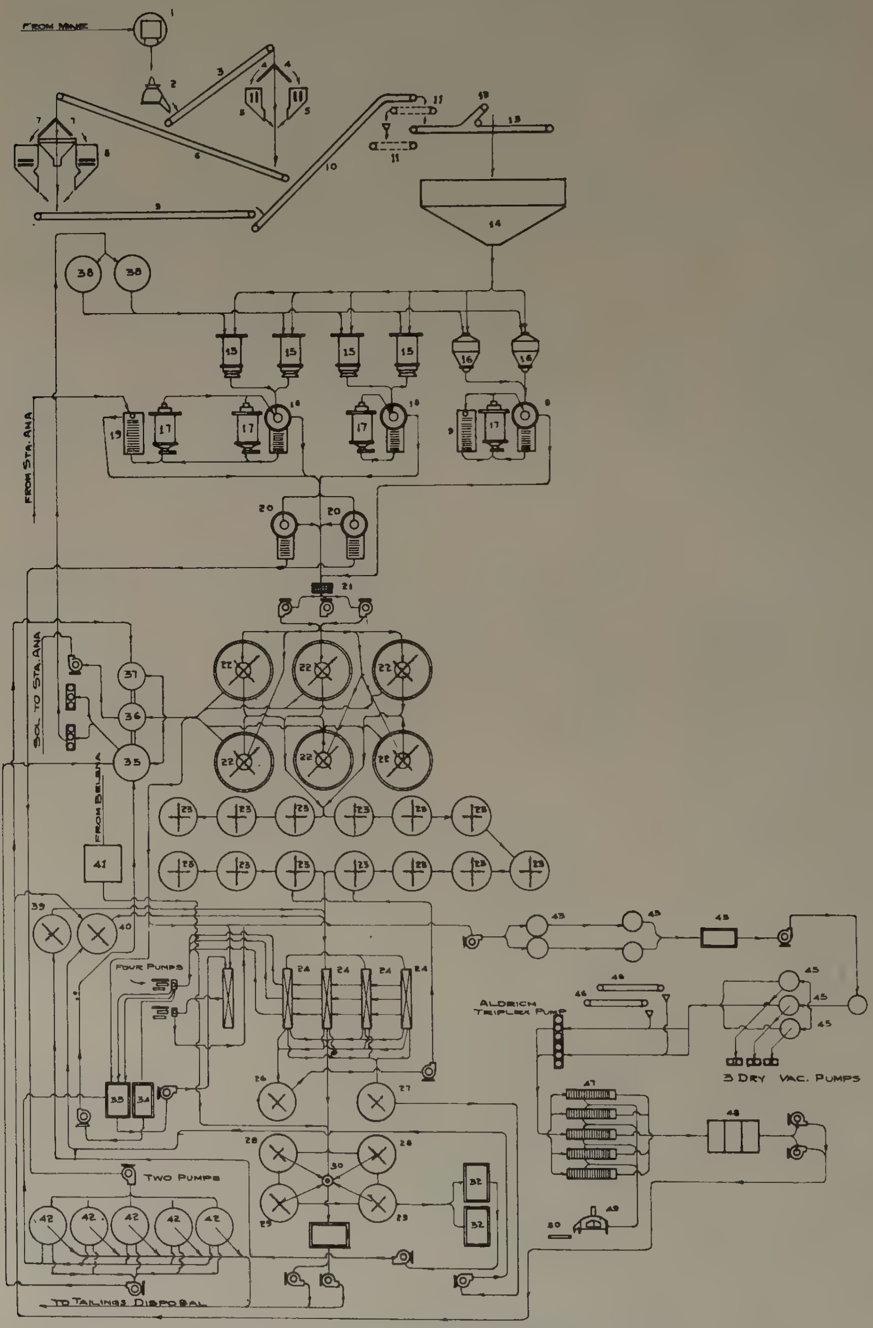

FIG. 1.—FOR CAPTION SEE OPPOSITE PAGE.

bottom and flows into a sealed roller spray chamber (No. 5) of wooden construction, 4 ft. 6 inches wide, 6 ft. high and 16 ft. long. The two rollers in this chamber revolve at 1000 r.p.m. and are set so that they dip 1 in. into the pulp as it flows through the chamber. The rollers throw this pulp into a finely divided spray, dispersing it so thoroughly throughout the chamber that it is in a condition favorable to the absorption of the circulating SO_2 gas. The pulp emerging from the roller spray chamber at the other end also flows into a conditioner (No. 6). From this tank the pulp flows by gravity to a 4-in. Wilfley centrifugal pump (No. 7) which elevates it to the top of the third tower (No. 8) where again it comes into contact with SO_2 gas, which, in this tower, is of the maximum strength produced by the roasting furnaces. From the bottom of this tower the pulp flows to the second conditioner tank (No. 9). At this stage of its treatment the pulp is in its state of highest acidification, having absorbed in passage through the towers and roller spray chamber all the SO_2 gas possible under the existing conditions of pulp dispersion, gas strength, velocity of gas and tonnage of pulp.

Before continuing with the further treatment of the pulp, it is advisable to trace the flow of SO_2 gas. This gas is produced by the roasting of pyrites supplied by the flotation plant. The furnaces (No. 17) are of the Herreshoff type. They are 8 ft. 6 in. in diameter and have seven hearths—the upper hearth serving as a drying hearth for the moist concentrate. The gas is drawn from the furnaces by either one of two Buffalo exhausting fans (No. 19), first through a large double-compartment dust chamber made of sheet steel (No. 18), which cools the gas and deposits the dust and condensed arsenic fumes before the fan is reached. On the discharge sides of the fan the gas is delivered, through a wooden pipe (No. 20), which is 16 in. in diameter (inside measurement), wire-wrapped and thoroughly asphalted to prevent leakage, to a point near the SO_2 towers, where sufficient gas is diverted by means of a conduit

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FIG. 1.—GENERAL FLOW SHEET, FRESNILLO MILL.

1. 10-ton steel car on rotating car dumper.
2. 30-in. McCully gyratory crusher.
3. 36-in. belt conveyor, 250 f.p.m.
4. Grizzly.
5. 48-in. horizontal Symons disk crushers.
6. 30-in. belt conveyor, 250 f.p.m.
7. 4 × 6 Leahy vibrating screen.
8. 48 Vert. Symons disk crushers.
9. 30 belt conveyor, 250 f.p.m.
10. 30 belt conveyor, 250 f.p.m.
11. Sample bucket conveyor.
12. Robins tripper.
13. 30-in. belt. conveyor, 250 f.p.m.
14. Ore bin. capacity 8800 tons.
15. 6 × 12 Marcy rod mill.
16. 8 × 48 Hardinge ball mill.
17. 6 × 14 Traylor ball mill.
18. 8 × 25 × 12 dia. Dorr bowl classifier.
19. 8 × 25 duplex classifier.
20. 6 × 26 × 15 dia. bowl.
21. Screen.
22. 60 × 10 Dorr thickener.
23. 40 × 25 Dorr agitator.
24. 10 × 56 Butters filter, 156 leaves per box.
25. 10 × 56 Butters clarifier, 156 leaves per box.
26. 36 × 12 excess pulp tank.
27. 36 × 12 excess barren-solution tank.
28. 36 × 12 dewatering tanks (final tailings).
29. 40 × 12 dewatering tanks (final tailings)
30. 4-in. Dorrco pumps.
31. Final-tailings sump.
32. Water storage.
33. Rich-solution sump.
34. Weak-solution sump.
35. 26 × 12 mill-solution tank.
36. 24 × 12 thickener-solution tank.
37. 24 × 12 barren-solution storage.
38. 36 × 16 mill-solution tank.
39. 36 × 12 water storage.
40. 36 × 12 barren-solution storage.
41. Water-control tank.
42. 60 × 16 sand leaching tanks.
43. Pregnant-solution tanks.
44. Steady-head steel tank.
45. 6 × 12 Crowe process vacuum receiver
46. Zinc-dust feeder.
47. Merrill filter presses.
48. Barren-solution storage.
49. Reverberatory smelting furnace.
50. Bullion casting wheel.

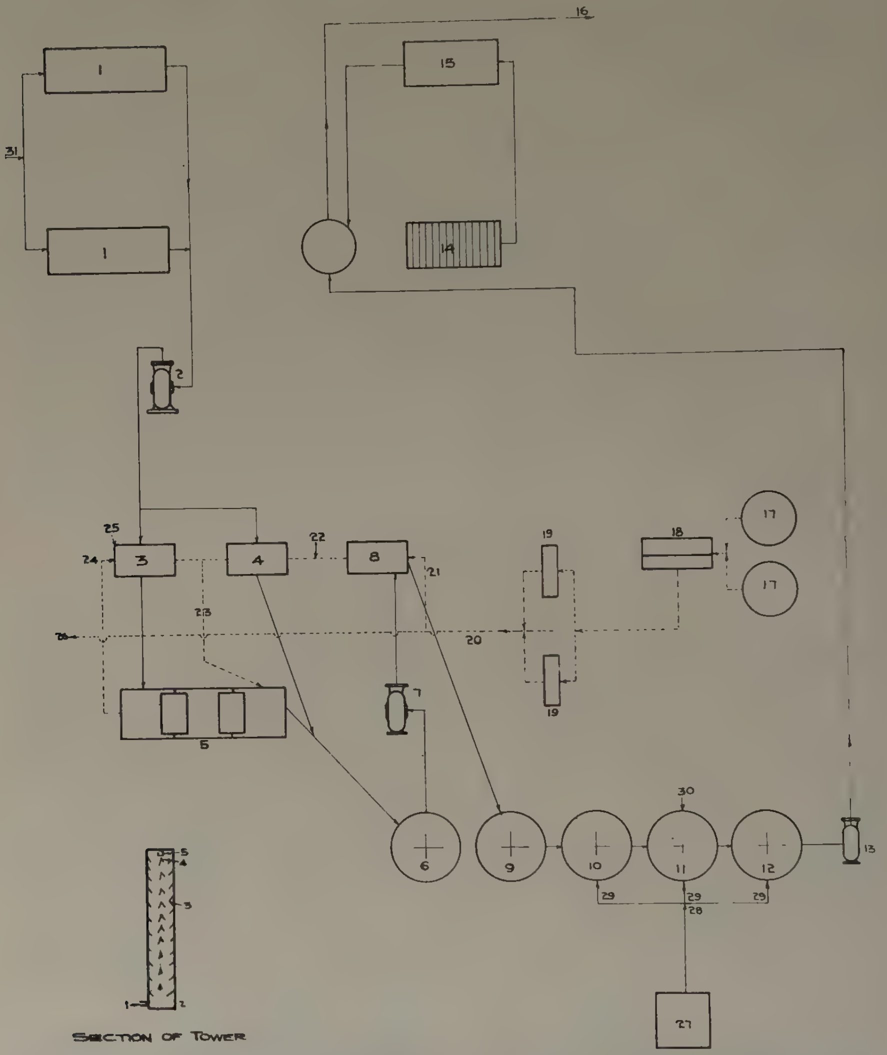

FIG. 2.—FLOW SHEET OF MANGANESE-SILVER SULFUR DIOXIDE TREATMENT PLANT AND SECTION OF TOWER.

FLOW SHEET

1. Marcy mill.
2. 7–13. Wilfley pump, 4 in.
3. 4–8. SO_2 absorption tower.
5. Roller spray chamber.
6. 9–12. Conditioner tank.
14. Dorr bowl classifier.
15. Traylor mill.
16. Pulp launder to cyanide plant.
17. Roasting furnace.
18. Dust chamber.
19. Exhausting fan.
20. Main gas conduit.
21–25. Auxiliary gas conduit for SO_2 absorption towers.
26. Gas conduit to cyanide recovery plant
27. Low-pressure blower.
28. Low-pressure air main.
29. Low-pressure manifold.
30. Lime-emulsion pipe.
31. "Spent"-solution pipe.

SECTION OF TOWER

1. Gas entrance.
2. Gas-seal pulp discharge.
3. Baffles.
4. Pulp spillage baffles.
5. Pulp launder.

(No. 26) to the cyanide recovery plant to meet the requirements of the process; the remaining gas enters at the bottom of the third tower (No. 8) at an average pressure equivalent to a head of 4 in. of water. The part of the gas not absorbed by the pulp, together with the residual air, discharges into a conduit (No. 22) into the top of the tower, and then to the bottom of the second tower (No. 4). About 50 per cent of the SO_2 gas entering the tower (No. 8) is absorbed in its passage. In the same manner the gas passes upward through the second tower (No. 4) where about 40 per cent of the remaining SO_2 is absorbed, to enter a conduit (No. 23), which delivers it to one end of the roller-spray chamber. After passing through this chamber the flow is through a conduit (No. 24) to the bottom of the first tower (No. 3). Passing upward through this tower the gas is discharged by a conduit (No. 25) into the atmosphere. In both the roller-spray chamber and tower (No. 3) about 50 per cent of the SO_2 remaining in the gas mixture is absorbed. The final result is the gas exhausting to the atmosphere, containing slightly over 1 per cent of SO_2, which indicates a total absorption of 85 per cent of the available SO_2. Thus it may be seen that the flow of pulp and SO_2 gas is countercurrent; the pulp being constantly enriched in acidity and the gas mixture being progressively depleted of SO_2 from unit to unit.

Referring again to the cycle of pulp treatment, there are five conditioner tanks, 16-ft. diameter by 11 ft. 6 in. high, all of which are arranged in series. The tanks are of the Devereux type, the impeller revolving at 100 r.p.m. The time of agitation in each tank is approximately 2.4 hr. when treating 330 tons per day and 1.2 hr. when treating 660 tons per day, the latter being considered the maximum allowable tonnage for the unit. From tank 9 the pulp flows continuously through tanks 10, 11 and 12. Each of these tanks has a manifold of small pipes (No. 29), dipping 6 in. below the surface of the pulp. These pipes are connected to a low-pressure air main (No. 28) into which air at 6 oz. of pressure is supplied by a blower (No. 27). In tank 10 this air serves to dissipate into the atmosphere the excess SO_2 gas in the pulp, which otherwise would require additional lime to neutralize its acidity. In tank 11 an emulsion of lime (No. 3) is added for the purpose of precipitating the dissolved manganous and ferrous compounds, as manganous and ferrous hydrates. The low-pressure air in this and the final tank (No. 12) assists in oxidizing the manganous and ferrous compounds to manganic and ferric compounds. After passing through tank 12 the pulp is returned to the mill by a 4-in. Wilfley centrifugal pump (No. 13) for regrinding. This is accomplished by discharging the pulp into a Dorr bowl duplex classifier (No. 14) set in closed circuit with a 6 by 14-ft. Traylor ball mill (No. 15). The overflow (No. 16) of this classifier, which averages 60 per cent minus 200-mesh material, joins the feed of the slime-treatment plant.

The gain in extraction accomplished by the sulfur dioxide treatment varies considerably with different ores, but it appears to be in direct proportion to the amount of manganese dissolved by the SO_2 gas—approximately 7 grams of silver for every 0.1 per cent of dissolved manganese. An increased recovery by this treatment, of 25 grams of silver per ton, represented a substantial economic advantage, when silver was quoted at around $0.30 (U.S.A.cy.) per ounce.

The laboratory pilot test, carried out daily in conjunction with the plant treatment, often showed as much as 35 grams additional recovery of silver. On account of mixing the SO_2 treated slimes with the general mill slimes, it was difficult to check the actual additional recovery in the plant.

An interesting point is noted in connection with tests for the oxygen content of solution in the pulp, leaving the final treatment tank (No. 13) of this unit. This solution is entirely devoid of free oxygen; moreover, it required several hours vigorous agitation with air to satisfy the oxygen-consuming capacity, and to render it susceptible to the absorption of free oxygen. In view of this, it is quite possible that a separate cyanide circuit for these treated slimes would be a distinct advantage, especially if it were so designed that several hours of agitation and aeration could be given prior to the addition of cyanide.

At the present time this plant is not operating, the reason being that the manganese content of the general ore is now only 0.4 per cent—a percentage well below a critical limit for an economical operation.

It is to be noted that a cheap source of sulfur is essential when treating low-grade silver ores, and that the successful application is limited to manganiferous silver ores that do not contain much more than 1 per cent of manganese. Also that the pulp is of a gelatinous nature, owing to the presence of ferric and manganic hydrates and, therefore, more difficult to filter. At Fresnillo this condition does not slow down the filtering rate to a marked extent, in view of the fact that the mixed feed to the filter plant contains a relatively high percentage of normal pulp.

ACKNOWLEDGMENTS

The author wishes to express his gratitude and indebtedness to Mr. T. C. Baker, General Manager of The Fresnillo Company at Fresnillo, for his assistance and advice in preparing this paper, and to Mr. H. A. Burk, Cyanide Plant Superintendent of The Fresnillo Company at Fresnillo, for his very valued help in collecting data for this paper.

Milling Methods at the Concentrator of The Fresnillo Company

By W. A. Binsacca* Member A.I.M.E.

The concentrator treats sulfide ore containing lead, copper, zinc, gold and silver, at the rate of 850 tons per 24 hr., and produces lead, copper, zinc and iron concentrates by selective flotation methods.

The ore occurs below the oxidized zone along a main fracture, the wall rock of which is either greywacke or calcareous shale. The predominating sulfide minerals are galena, sphalerite, pyrite and chalcopyrite; the others are quartz and calcite. In minor amounts, arsenopyrite, pyrrhotite and hematite occur, and such secondary minerals as smithsonite, chalcocite, covellite, bornite, cerussite and anglesite are detected in the ore, the last two named occurring principally as surface alterations of the galena.

The ore is hauled from the mine bins in 10-ton cars which pass over the track scales before entering the car dumper ahead of the crusher plant.[1] A conveyor belt delivers the crushed ore to the sulfide bin, which has a capacity of 2800 tons.

The primary grinding unit consists of two 6 by 12-ft. Marcy rod mills operating in parallel and in open circuit, each taking 425 tons per day. One rod mill discharges into an 8 by 25-ft. Dorr classifier with a 12-ft. bowl and the other into a 6 by 25-ft. Dorr duplex classifier. These classifiers are arranged so that each unit rakes the sands into a 6 by 14-ft. Traylor ball mill, which discharges to the bowl classifier only. The duplex classifier therefore operates in open circuit and the bowl classifier in closed circuit, the overflow of both classifiers furnishing the feed for the flotation section. Other groupings have been tried, but this has been found the most satisfactory.

The Marcy mills are driven by 150-hp. slip-ring induction motors with speed reducers to give 17 r.p.m. The Traylor mill operates at a speed of 24 r.p.m. and is driven by a 200-hp. slip-ring induction motor, direct connected to pinion and gear. The bowl classifier operates at a speed of 22 r.p.m., with the bowl rake making 4.5 r.p.m. and the duplex classifier at 31 r.p.m.

High-carbon steel rods of Mexican manufacture, 12 ft. long by 3-in. diameter, are used in the Marcy mills, with a resulting consumption of 1.52 lb. per ton ore. The Traylor mill is fed 3-in. cast-iron balls cast in

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[1] See W. E. Crawford, page 734, this volume.

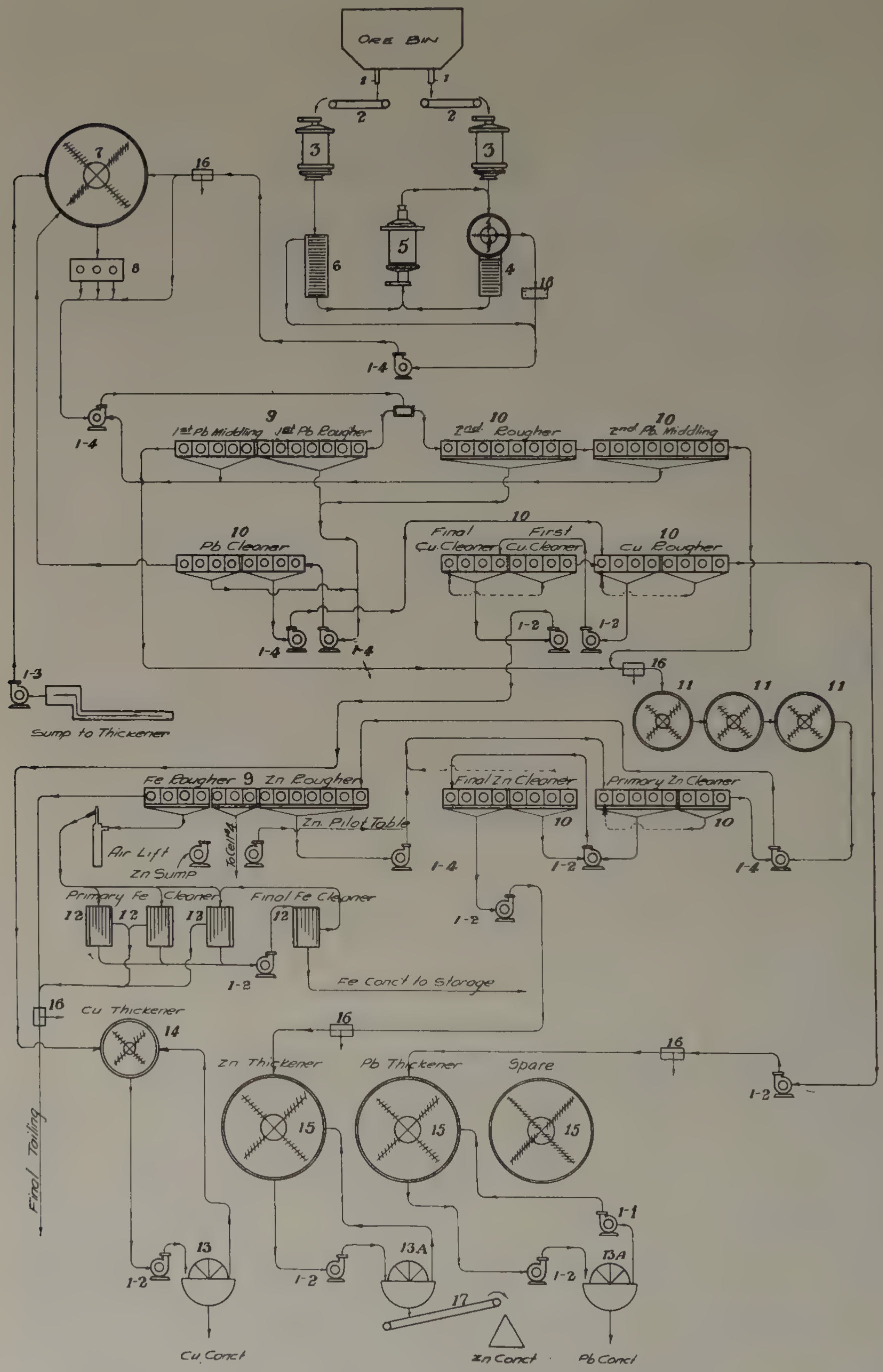

FIG 1.—FOR CAPTION SEE OPPOSITE PAGE.

the local foundry, and the consumption is 1.04 lb. per ton of ore treated. The Traylor mill is lined with manganese-steel liners made in Mexico; consumption amounts to 0.14 lb. per ton ore. The Marcy mills have a lining of locally cast molybdenum-chrome steel, but consumption data are not available at this time.

The primary grinding was formerly done in two 8-ft. by 48-in. Hardinge ball mills, but since September, 1933, the sulfide ore has been delivered to one of the Marcy rod-mill units, leaving the Hardinge mills available for oxide ore. By the change it was possible to increase the tonnage from 750 to 850 tons per day, but the fineness of the finished product dropped from 65 to 54 per cent minus 200 mesh. The relative mechanical efficiencies of the units were unchanged, but since equally good results were obtained in the flotation plant on the coarser product, an increase in capacity was obtained without increasing grinding costs. On the other hand, the oxide operation also benefited by the fine grinding of the Hardinge mills.

The pulp in the Marcy mills averages between 76 and 79 per cent solids, and in the classifier overflow between 40 and 42 per cent solids. The screen analyses of the mill products are given in Table 1.

The classifier overflow is passed through a revolving sheet-iron screen 3 ft. in diameter by 6 ft. long, with ¼-in. perforations to remove the chips. The operation is simple and indispensable where pulp containing chips is handled with pump and flotation-machine impellers of the closed type. The pulp is elevated with a 4-in. Wilfley sand pump to a distributor, which diverts 60 per cent of the flow to a 60-ft. Dorr thickener for dewatering. Three Dorrco diaphragm pumps discharge the thickened pulp at 50 per cent solids to a launder, where it joins the bypassed pulp to flow to a 4-in. Wilfley pump for elevation to the head of the lead section.

Conditioning ahead of the lead section was abolished several years ago. All reagents for the lead float are added at the pump box. The lead middling joins the original flow at this point, diluting the pulp to 42 to 44 per cent solids.

The lead roughing section consists of two units in parallel. One is a 12-cell, 24-in. Minerals Separation sub-A machine driven through V-ropes taking 70 per cent of the feed, and the other is composed of two 8-cell, 18-in. gear-driven machines of the same make connected in series.

FIG. 1.—FLOW SHEET OF THE FRESNILLO CONCENTRATOR.

1. 8-in. pipe chute feeders.
2. 18-in. conveyor belts.
3. 6 by 12-ft. Marcy rod mills.
4. 8 by 25-ft. Dorr duplex 12-ft. bowl classifier.
5. 6 by 14-ft. Traylor ball mill.
6. 6 by 25-ft. duplex classifier.
7. 60-ft. Dorr thickener.
8. 4-in. Dorrco diaphragm pumps.
9. 24-in. Minerals Separation flotation machine.
10. 18-in. Minerals Separation flotation machine.
11. 8 by 6-ft. Devereux agitators.
12. Standard Plat-o concentrating tables.
13. 6-ft. dia. American continuous 2-disk filter.
13a. 6-ft. dia. American continuous 4-disk filter.
14. 15-ft. thickener.
15. 40-ft. Dorr thickener.
16. Automatic sampler.
17. 16-in. belt conveyor.
18. 3 by 6-ft. revolving chip screen.
1–1. 1-in. Wilfley pumps.
1–2. 2-in. Wilfley pumps.
1–3. 3-in. Krogh pumps.
1–4. 4-in. Wilfley pumps

TABLE 1.—*Screen Analyses of Fresnillo Mill Products*

Screen Size	Crusher Product		Rod-mill Discharge		Classifier Overflow	
	Weight Per Cent	Cumulative Weight Per Cent	Weight Per Cent	Cumulative Weight Per Cent	Weight Per Cent	Cumulative Weight Per Cent
Inch						
On 1.00	6.0	6.0				
0.75	17.1	23.1				
0.50	18.6	41.7				
0.375	9.3	51.0				
Mesh						
On 3	7.4	58.4				
4	5.5	63.9				
6	3.7	67.6	None	None		
8	3.2	70.8	0.2	0.2		
10	3.7	74.5	1.0	1.2		
14	2.1	76.6	2.4	3.6		
20	2.3	78.9	5.4	9.0		
28	2.2	81.1	8.4	17.4	None	None
35	1.9	83.0	10.6	28.0	1.1	1.1
48	1.8	84.8	9.9	37.9	6.6	7.7
65	1.9	86.7	9.1	47.0	8.9	16.6
100	1.8	88.5	9.3	56.3	10.2	26.8
150	1.7	90.2	7.9	64.2	11.6	38.4
200	1.1	91.3	8.3	72.5	8.0	46.4
Through 200	8.7	100.0	27.5	100.0	53.6	100.0

The rougher concentrate containing lead, copper, gold and silver is produced from the first seven cells of the 24-in. machine and from the first 18-in. machine. The remaining cells produce a middling, which is laundered to the pump feeding this section. The rougher concentrate is elevated through a 4-in. Wilfley pump to an 8-cell, 18-in. Minerals Separation sub-A machine for cleaning. The tailing is laundered to the 60-ft. thickener at the head of the circuit; the cleaned lead-copper concentrate drops to a 4-in. Wilfley pump. Here the sodium dichromate is added and the charge is elevated to a 12-cell, 18-in. V-rope-driven Minerals Separation sub-A machine, for the separation of the bulk lead-copper concentrate into separate lead and copper concentrates. The froth from this machine is a rougher copper concentrate, which is cleaned twice in an adjoining 8-cell, 18-in. gear-driven machine to produce a finished copper concentrate and a tail, the latter returning to the head of the 12-cell machine. The tail of this 12-cell machine is the finished lead concentrate. The operation of the lead-copper separation section is simple and easily controlled by adjusting the quantity of froth with the amount of air. The optimum condition is one that gives a minimum amount of lead in the copper concentrate and the least amount of copper in the lead concentrate. Care must be taken that none of the dichromate-treated pulp gets back into the lead circuit, since even small quantities are detrimental in the flotation of galena.

To three Devereux tanks, 8 ft. in diameter by 6 ft., is laundered the tailing from the lead section. These tanks are in series and serve as conditioners for the zinc circuit. To the first is added the cyanide, to the second the copper sulfate, and to the 4-in. Wilfley pump at the end of the third tank are added the xanthate and oils. The conditioned pulp is elevated to a 14-cell, 24-in. V-rope-driven Minerals Separation sub-A machine. The froth from the first eight cells is the rougher zinc concentrate; the next two cells produce a middling, which returns by gravity to the fourth cell. The rougher concentrate is elevated with a 4-in. Wilfley pump to the first of two 8-cell, 18-in. gear-driven machines of the same make for double cleaning. These two machines produce a finished zinc concentrate and a cleaner tail, the latter flowing by gravity to the pump to the head of the zinc section.

The tailing from the zinc rougher section passes to the last four cells of the machine, where more oil and xanthate are added; and a rougher iron concentrate is pulled for its gold and silver contents. The tail from the iron section is discarded. The iron rougher concentrate is passed over three Deister-plato tables in parallel and the concentrate recleaned on a fourth table. The table tailing containing fine slimes and coarse gangue joins the rougher iron tailing to constitute the final plant tailing. It is laundered to the dewatering tanks of the cyanide mill and pumped with the cyanide plant tailing to the tailings dam.

Pumps deliver the lead concentrate to a 40-ft. Dorr thickener and the underflow of the latter to a 4-disk, 6-ft. American filter. The filter drops a cake containing 12 per cent moisture into ½-ton buggies, which are emptied on a platform for air drying. Under ordinary atmospheric conditions the concentrate is thus reduced to approximately 6 per cent moisture before shipping. The copper concentrate is handled in the same manner, except that the thickener is 15 ft. in diameter and the filter has two disks. This cake has a moisture of 14 per cent. The zinc concentrate is thickened and filtered in the same way as the lead concentrate, but the filter cake, containing 7 to 8 per cent moisture, is stacked in a conical pile by a conveyor belt, which elevates the concentrate to a height of 40 ft. The iron concentrate is laundered from the last table to settling ponds. Part of this concentrate is shipped direct to the smelter and part is roasted for sulfur dioxide for use in the cyanide recovery plant, an adjunct of the cyanide plant. The sinter, assaying approximately 0.08 oz. gold and 22 oz. silver per ton, is then shipped to the smelter. The lead, copper and iron concentrates are smelted in Mexico, while the zinc concentrate is shipped monthly to European ports.

TABLE 2.—*Consumption of Reagents*

Place Added	Reagent	Strength Solution, Per Cent	Pounds per Ton Ore Treated
Lead section	Zinc sulfate	17.0	0.700
	Sodium cyanide	2.5	0.026
	Ethyl xanthate	10.0	0.254
	Cresylic acid		0.156
	Barrett No. 634		0.167
	G. N. S. pine oil		0.014
Lead-copper separation	Sodium dichromate	10.0	0.276
	Zinc sulfate	17.0	0.200
Zinc section	Sodium cyanide	2.5	0.074
	Copper sulfate, saturated		0.630
	Ethyl xanthate	10.0	0.018
	Barrett 634		0.102
	G. N. S. pine oil		0.008
Iron section	Ethyl xanthate	10.0	0.108
	Barrett 634		0.026
	G. N. S. pine oil		0.002

All reagents, except the copper sulfate, are added from a centrally located eight-compartment feeder of the cup and disk type. The copper sulfate is added as a saturated solution, by passing a measured stream of water through a concrete box full of the crystals.

The cyanide is added to depress pyrite, and it is kept at a minimum in the lead section inasmuch as cyanide also has a slight depressing effect on the copper and silver minerals. At the zinc section, it is added to the heads of both the rougher and cleaner circuits. Cyanide depresses the pyrite, but with no effect on the sphalerite after it has been activated with copper sulfate.

The fairly high consumptions of xanthate and oil in the lead circuit are due to their absorption by the carbonaceous shale in the ore.

The sodium dichromate renders the galena unfloatable in the lead-copper separation section by forming an oxidized film on that mineral. The zinc sulfate is not essential for this separation, but is added to keep the zinc in the copper concentrate below the smelter penalty limit.

The mine water has a temperature of 75° to 85° F. and is alkaline, having a pH of 7.7 to 7.9. The ore is also alkaline and when ground in distilled water the pH of the water rises to 8.0. In 1929 the addition of an alkaline salt was discontinued, with no effect on the metallurgical results.

Typical grades of plant products and recoveries are given in Table 3.

The fineness of the flotation feed has been gradually reduced from 1.0 per cent on 65 mesh and 80 per cent minus 200 mesh at the beginning

TABLE 3.—*Typical Grades of Products and Recoveries*

Product	Per Cent Weight	Au, Oz.	Ag, Oz.	Pb, Per Cent	Cu, Per Cent	Zn, Per Cent	Fe, Per Cent	nsol., Per Cent
Head	100.0	0.013	10.7	7.9	0.71	8.3	7.6	55.0
Pb-Cu bulk	13.16	0.047	59.5	56.3	4.30	6.7		
Lead concentrate	10.60	0.029	45.3	68.0	0.70	4.6	4.1	2.6
Copper concentrate	2.56	0.119	117.5	7.0	18.00	12.9	21.0	6.8
Zinc concentrate	12.40	0.010	10.6	0.8	0.70	52.0	10.8	1.2
Iron concentrate	3.50	0.064	16.7	0.7	0.50	8.0	36.7	3.3
Final tailing	70.94	0.004	1.4	0.5	0.07	1.0	5.9	

RECOVERIES, PER CENT

Product	Au	Ag	Pb	Cu	Zn
Head	100.0	100.0	100.0	100.0	100.0
Lead-copper bulk	47.8	73.0	93.8	79.0	10.0
Lead concentrate	23.8	45.0	91.5	10.4	6.0
Copper concentrate	24.0	28.0	2.3	68.6	4.0
Zinc concentrate	9.2	12.1	1.3	12.1	78.0
Iron concentrate	17.5	5.4	0.3	2.4	3.4
Final tailing	25.5	9.5	4.6	6.5	8.6

of operations in 1925 to the present 17 per cent on 65 mesh and 54 per cent minus 200 mesh. The metallurgy as a whole has not suffered by the coarser grind. The minerals in the ore break relatively free in sizes through 100 mesh, and while locked minerals appear in the coarser feed, the amount so locked is small. The lead losses are in the finer sizes, and it was brought out by microscopic research that the major part of these losses is due to the oxidation of the surfaces of the galena particles. Galena in freshly mined ore is relatively free of oxidation, but after coming in contact with water in the stopes the oxidation is rapid, due very likely to the effects of the salts in the mine water.

Practically all of the chalcopyrite remaining in the tailing of the lead section is in the form of minute inclusions in the sphalerite. These inclusions are so small that a separation by finer grinding could not be made economically. The sphalerite is in the form of a marmatite, is dark brown in color, and contains roughly 10 per cent of iron. A clean zinc concentrate free of pyrite will contain not more than 53 per cent of zinc. The insoluble in the lead and copper concentrates is present as a fine slime of carbonaceous shale. This slime floats readily in the lead section, hinders flotation, and is the cause of the high moisture in the dewatered concentrates. With the present coarse grind less of this shale is slimed.

The control of the plant by the operators is based on the appearance of the froth and by use of the vanning plaque. A small portion of the final zinc concentrate passes over a pilot table. A streak of galena denotes poor work in the lead section, and a streak of pyrite warns the operators that the grade of the zinc concentrate is low. Simplicity and ease of operation have been the objectives of all changes and additions in the plant, and adherence to this policy has made for consistently uniform results. The plant operates six days a week; all major repairs are made when the plant is down. Shutdowns during the operating period are extremely rare.

The plant is situated on a flat site, with the grinding mills and flotation cells on the same level. The grinding, the lead, the lead-copper separation, and zinc sections each have a separate clean-up sump, so that one section is not contaminated by the spills from another. In emptying a sump, the product is pumped to the head of its respective section, except the lead section clean-up, which goes first to the 60-ft. thickener for dewatering.

The sand pumps for handling the pulps are either 2-in. or 4-in. Wilfleys. Important and fast wearing pumps are installed in duplicate. The wearing parts are made of white iron and last 1½ to 6 months, depending on the product elevated.

One No. 3 Roots blower, operating at 410 r.p.m. and requiring 15 hp., supplies 1400 cu. ft. of air per minute at a pressure of 1.75 lb. per sq. in. for the 86 flotation cells.

The Minerals Separation machines have white-iron impellers of the closed type. The life of the impellers in the roughing circuits ranges from 8 to 12 months, and in the concentrate cleaning circuits from 12 to 16 months. Baffles are not used in the cells, the swirl being broken by one layer of grids supported on two wooden cross pieces. The bottom and side liners are made of white iron and have a life of several years.

Various types of pneumatic flotation machines have been operated, but they have all been discarded in favor of the mechanical type, which produces better metallurgical results on this class of ore. The air machines required higher quantities of frothers, were more sensitive to variations in the ore, and would not operate as uniformly on coarse feed.

The head sample for the concentrator is taken by a Geary-Jennings automatic sampler, which takes a cut every 12 min. of the classifier overflow. A similar sampler is also used for the tailing sample. Other samples are cut at regular intervals by hand or by means of water-tipping devices.

The water supply is derived from the bottom levels of the mine. A pump delivers the water from the surface reservoir to the mill tank and to the various sprays and water lines in the plant. The mill tank also receives the overflow of the 60-ft. pulp thickener and of the zinc concentrate thickener. The overflows of the lead and copper concentrate thickener and the filtrates of the American filters are contaminated with sodium dichromate; consequently they are discarded to the cyanide-plant dewatering tanks. Water recovered from the tailings dam is contaminated with cyanide and lime and is not used in the flotation plant.

TABLE 4.—*Distribution of Power Consumed per Ton Ore Treated*

ITEM	KILOWATT-HOURS
Crushing and conveying	1.78
Grinding and classifying	9.90
Flotation machines, blower, dewatering, filtering concentrates and tabling	6.80 (not segregated)
Pumping	3.09
Water supply	2.55
Disposal of tailing	0.30
Total	24.42

The foregoing covers the general practice in treating what is locally considered a normal type of complex sulfide ore. However, recently the company has been treating sulfide ores from outside sources and is developing a light type of sulfide ore at Fresnillo, and these require a somewhat different treatment. To meet this situation, a 4-cell, 24-in. Minerals Separation sub-A machine was placed in operation. The feed for this new section is ground with a 4 by 13-ft. Krupp ball mill in closed circuit with a 42-in. by 20-ft. simplex classifier. Ore is now treated in this section at the rate of 85 tons per day, but the tonnage will be increased, when conditions warrant, to 170 tons per day by placing in operation a second Krupp ball mill of the same size. A bulk gold-silver-lead-iron concentrate is produced in this new section, and the concentrate is pumped to the lead concentrate thickener, where it mixes with the lead concentrate from the main plant. The tailing is diverted to the zinc section of the main plant and a small amount of zinc recovered from it. The advantage of the bulk float is that gold and silver recoveries are increased by floating the pyrite and zinc. The latter incurs no penalty, since the zinc content of the ore is low, and since the tonnage of bulk concentrate is relatively small compared to the total production of lead concentrate. This unit is not shown in Fig. 1.

ACKNOWLEDGMENT

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Development of Milling and Cyanidation on the Witwatersrand

By F. Wartenweiler,* Member A.I.M.E.

Much has been written on milling and cyanidation as practice on this gold field, therefore the purpose of this paper is to record its development only briefly and to describe in more detail the contemporary state of the science and art of this branch of metallurgy. It is a pleasing task, because a steady evolution is to be recorded.

Definite trends influenced the development. Some have the stamp of original thought and observation; many have been achieved from a logical sequence of planned experimentation and research; while others have resulted from the drive of sheer necessity. The large scale of operations and capital outlay has compelled conservative progress.

Banket Ore

For a better understanding, a review of the characteristics of the ore may not be out of place. The Witwatersrand gold ore is named "banket" from the similarity of the oxidized outcrop ore to the Boer almond cake of that name. The interbedded orebody is commonly called "reef." It is a conglomerate composed of various sizes of pebbles, principally non-gold-bearing white quartz, which constitute about 70 per cent of the bulk, within a matrix of cementing material consisting of fine-grained quartz, sericite, chlorite and chloritoid and containing the precious metals and mineral sulfides. Argillaceous components in the mill pulp may be as high as 32 per cent. Of the sulfide minerals, pyrite and pyrrhotite are predominant. Most base-metal sulfides are present, but sporadically and generally only found in highly concentrated mill products. In the latter, osmiridium is recovered in varying amounts; from 1 oz. in 3000 tons to 1 oz. in 10,000 tons of ore. Most of the oxidized ore has been mined, so milling is confined principally to the sulfide ore.

A typical analysis of sulfide ore is: silica (SiO_2), 86.76 per cent; pyrite (FeS_2), 2.75; ferric oxide (Fe_2O_3), 2.65; alumina (Al_2O_3), 6.91; lime (CaO), tr.; magnesia (MgO), 0.70; sp. gr., 2.7.

Milling grade at present price of gold (137 shillings per ounce) varies from 3.5 to 8.5 dwt., with one mine milling ore as high as 16 dwt. The

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gold-silver ratio is in the neighborhood of 9:1. Other characteristics are: a high degree of abrasiveness; with depth (and stoping is proceeding as low as 7500 ft., vertical depth), it becomes denser and, owing to the high rock pressure, more brittle.

The gold in the ore may be divided into the portion that is in a free metallic state, and the portion that is in the same state but closely associated with the pyrite, being found within and between crystal faces. A recent laboratory investigation of a central district ore discloses the following distribution:

1. On being ground to 92 per cent −200 mesh: in the free state, 47 per cent; encased in gangue, 9 per cent; associated with pyrite minerals, 44 per cent.
2. On being ground to 92 per cent −200 mesh: dissolved in cyanide, 97 per cent.
3. On being ground to 50 per cent −200 mesh: subject to recovery by simple blanket concentration, 33.0 per cent.

Research carried out some years ago showed the size of typical gold particles to be as given in Table 1.

TABLE 1.—*Sizes of Typical Gold Particles*

District	Percentages of Sizes				
	+60 Mesh	−60 +90	−90 +200	−200 +260	−260
	400–700 Microns	200–400 Microns	100–200 Microns	76–120 Microns	10–70 Microns
Eastern	3.44	6.29	16.00	10.71	63.56
Central	2.30	10.90	20.60	19.50	46.70

The gold is irregular in shape with generally rounded contours. A large part, in its natural state, is sufficiently small to be rapidly dissolved in cyanide solution.

The reef is generally a clearly defined stratum, bounded by sedimentary walls of barren quartzite or by quartzite and shale. Several parallel reefs are payable, differentiating along the 60-mile Witwatersrand formation at present being mined.

Gradations are from a mere film of gold at a parting to a thickness of one inch of some "leader," to 6 ft. and more in other reef sections. As a stoping width of 36 in. is about a minimum in the flatter sections, it becomes apparent that considerable non-gold-bearing wall rock, particularly of quartzite, is broken in the course of mining. This is sometimes broken separately, as in resuing, and left underground, or according to circumstances it is broken at the same time as the reef and is partly sorted underground. A considerable amount reaches the surface as a mixture with the ore. In the course of mining, with water used in the drills and for dust allaying, a not inconsiderable amount of freed gold and pyrite are concentrated on the rough floor of the stopes. This, together with fine rock, is swept and washed to collecting points, whence it is sent to the reduction plants.

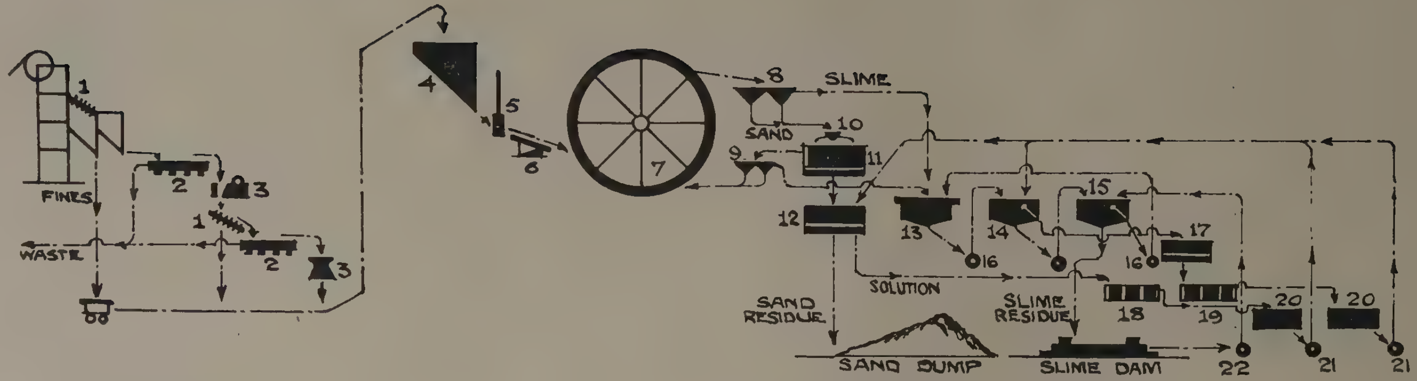
General Development

The early plants for ore reduction consisted of sorting tables, crushers, light California stamps and amalgamating plate tables, following common contemporary practice elsewhere. Amalgamation inside of the mortar boxes was also practiced. Amalgam barrels and batea were used in amalgamating trap concentrate and cleaning amalgam. Recovery was in the neighborhood of 75 per cent. With the depletion of the oxidized ore on the upper level, and the necessity for mining sulfide ore, gravity concentration with Frue vanners was added to the simple recovery scheme. The concentrates from various mines were treated by the then new chlorination process in a central plant, yielding a high recovery. The next advance was the treatment of the tailings with the cyanide process, the development of which is described later on.

The early crusher stations were comparatively complex, with two-stage crushing and sorting. Then came a simple phase, with one stage only, and this was continued until the period of the stampless plant, when there arose a need for a finer crusher product than was supplied to stamps. This led to two-stage crushing; the first with a jaw or gyratory crusher, and the second with disk crushers. More recently, the second stage of the work has been allotted to the newer types of fast-running gyratory crushers. Crushers placed underground were abandoned after a time, owing to difficulties in collecting their objectionable dust.

In view of the mining conditions, sorting becomes the first step in treatment of ore. It was practiced in the early plants by dumping the rock on a flat sheet, washing with a water spray and picking out the waste. Then came horizontal annular revolving tables, replaced today by belts with sorting in one or two stages, usually the former. Except where there is an enrichment at the reef contact, particularly at the shale wall or where gold is in scattered carbon particles, sorting can be done effectively.

The stamp mills rapidly developed with the largely increased tonnages mined, and the friendly competitive spirit among the millmen. Heavy California stamps of 2000-lb. dropping weight were the ultimate advancement. With experience and skilful engineering, many departures from the older design were brought about, such as cast-steel battery frames, wood battery posts reinforced with steel girders, multiple cam-shaft bearings, cast-steel anvil blocks and reinforced concrete mortar-box blocks.



1. Grizzlies
2. Sorting tables
3. Rock crushers, jaw or gyratory
4. Mill ore bin
5. Stamps
6. Amalgamation tables
7. Elevating wheel
8. Scalping spitzkasten, sand/slime
9. Return spitzkasten
10. Butters and Mein distributor
11. Sand collecting tank
12. Sand treatment tank
13. Slime collecting tank
14. First decantation tank
15. Second decantation tank
16. Pump services
17. Solution clarifier tank
18. Sand solution zinc boxes
19. Slime solution zinc boxes
20. Solution sumps
21. Solution pumps
22. Return solution pump

Fig. 1.—Composite flow sheet of practice from 1896 to 1904, prior to tube milling.

Features: Simplicity of flow; two-stage sorting and crushing; one pulp lift; superimposed sand tanks; decantation slime treatment; zinc-shaving precipitation.

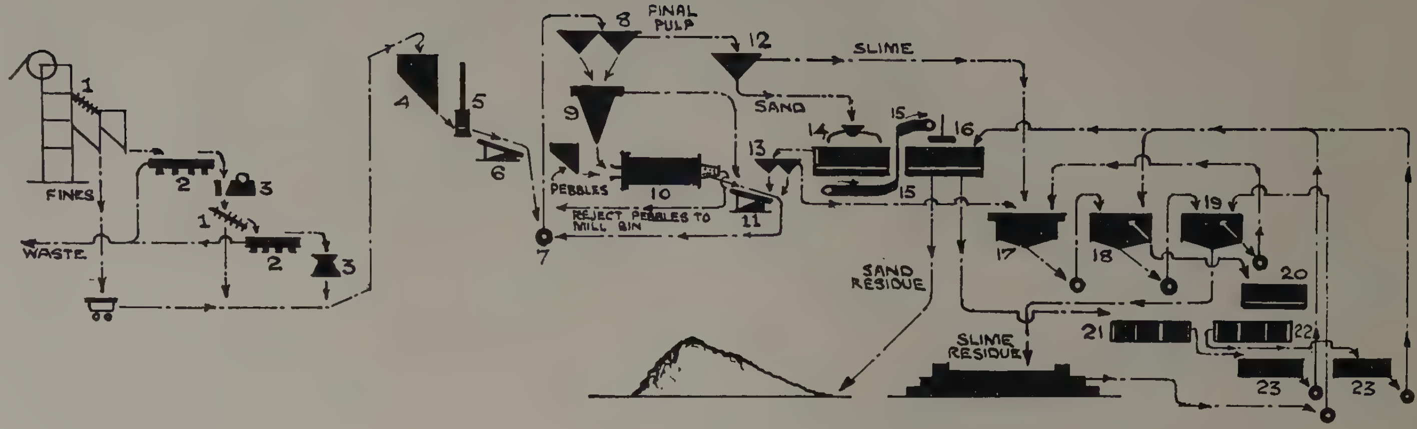
Of the heavy stamp mills running today, the best examples incorporate reinforced concrete mortar blocks, heavy mortar boxes either set on the concrete blocks with a felt mat between, or on an anvil resting on the concrete-block, wooden steel girder, reinforced battery posts resting on cast-iron shoes, cast stem guides and five-stamp units driven separately. With few exceptions, all mills are electrically driven. Mill structure and bin framing are of steel. Nissen stamps weighing one ton, with their separate mortar boxes, were first used in 1912 and are playing an important part in later mills.

Tube mills were introduced in 1904, the first being installed at the Glen Deep mill. Imported flint pebbles were the grinding media at first. Then the fortunate discovery was made that pieces of ore of a suitable size made excellent pebbles. These could be picked off the sorting belt. So ore pebbles have been in use since that time, and have influenced design and practice throughout. Of recent years, with the press for tonnage, a composite load of pebbles and steel balls has been adopted in a number of plants insufficiently equipped. In the development of tube milling, feeders with open bowls have displaced the circular scoop type, and positive discharge lifts have taken the place of the open discharge. A spent-pebble trommel is bolted to the discharge trunnion. The types of liners and their material have been legion, smooth wood blocks, reinforced concrete blocks, flint blocks, steel bars (Osborn liner) and cast-steel or iron sections. The last two have survived. The Osborn liner is a pebble packing liner, and the section liners are either packing or have a wave cross-section. The use of holding bolts has not found favor, and the common practice is to hold the liners with keys. The gold lock-up by a tube-mill liner in a water grinding circuit may be as high as 1000 oz. In a solution circuit it may be only one-tenth as much.

Corduroy blanket tables replaced amalgamating tables in 1923. For tube-mill classifiers, the development has been from plain thickening cones with nozzle discharge to Caldecott diaphragm thickening cones, to Dorr rake classifiers, Dorr bowl classifiers and spiral classifiers.

In size, the first tubes were 5 ft. in diameter and 20 ft. long; then the standards became 5 ft. 6 in. by 22 ft. and 6 by 16 ft., which have suited the local conditions admirably when serving as a complement to a stamp mill. With the introduction of plants without stamps, a larger size of tube (6 ft. 6 in. by 20 ft.) was preferred, and later mills 8 ft. in diameter by 16 ft. long were installed at two plants.

With experience, the adaptability of the tube mill was more fully recognized, and in 1920 trials on a working scale, carried out at Springs mines, demonstrated that the stamp could be dispensed with and the crushed ore fed directly to tube mills. This step is an important milestone in general milling development on this field, and is largely due to the



1. Grizzlies
2. Sorting tables
3. Rock crushers, jaw or gyratory
4. Ore bin
5. Stamps
6. Amalgamation tables
7. Sand pump
8. Spitzkasten
9. Dewatering diaphragm cone
10. Tube mill
11. Amalgamation tables
12. Sand/slime spitzkasten
13. Safety return spitzkasten
14. Sand collector with Butters and Mein distributor
15. Belt transfer of sand
16. Sand treatment tank with Blaisdell distributor
17. Slime collector
18. First decantation tank
19. Second decantation tank
20. Solution clarifier tank
21. Sand solution zinc boxes
22. Slime solution zinc boxes
23. Solution sumps

Fig. 2.—Composite flow sheet, 1904 to 1910, after introduction of tube milling.

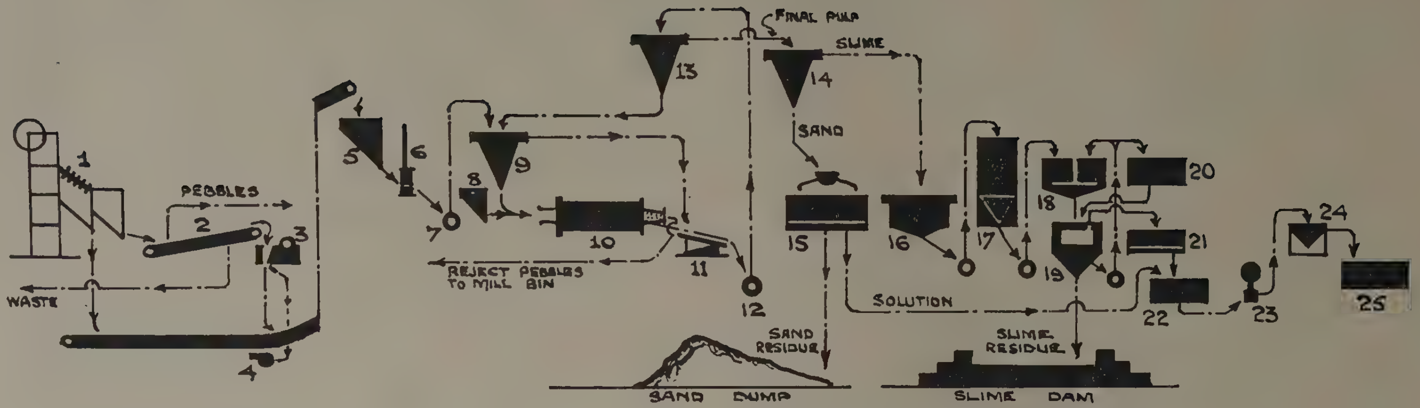
Features: Two-stage sorting and crushing; closed tube-mill circuit; use of pump instead of elevating wheel; belt sand transfer and Blaisdell distributor; decantation slime treatment; zinc-shaving precipitation.

work of Messrs. Willey and Ewing. The Springs plant was altered for its adoption. The first new plant to "go all out" for the idea was at New State Areas in 1923, where 13 large tubes were installed to take the ore direct from the secondary disk crushers and grind to a one-product pulp in one stage. Of more recent years, two-stage and three-stage tube-mill grinding is practiced: in the newer plants, the mills are served by classifiers of the rake, bowl and spiral type, which give a selective classification of pyrite, in conjunction with what is termed locally "bulk grinding."

The employment of ball mills was attempted in 1912, when the two-compartment "Gieseke" ball mill was given a trial on run of mine ore feed. The performance as regards grinding was satisfactory, but ball consumption on the abrasive ore proved prohibitive. Mill proportions and the principle of design were also at fault. More recently modern short, large-diameter ball mills with adequate classifiers have been installed at one mine.

The Witwatersrand can claim to have been a nursery for cyanidation, and the pioneer MacArthur and Forrest cyanide process for the recovery of gold and silver. From the first demonstration tank (in 1890), holding a 1¼-ton charge, with treatment by agitation in a potassium cyanide solution of a strength of 0.20 to 1.0 per cent, there has been an advance to agitating tanks holding a 500-ton charge in a sodium cyanide solution of strengths as low as 0.01 per cent, and also an increase in plants from a treatment capacity of a few thousand tons per month to the present-day (1934) single plant operation treating as high as 330,000 tons in the same period. Progress was rapid and within three years from the introduction 25 per cent of the total Rand production was from this source. Today this has mounted to over 50 per cent, and on several mines to practically 100 per cent. On the chemical side, progress in the early days was phenomenal, and many scientific and capable men were attracted here from various countries by the extraordinary opportunities offered in developing a new art and by the financial importance of improvements.

Cyanide treatment began with agitation of amalgamation tailing and of concentrate in wooden vats, the solution being decanted. From this, it developed to the use of superimposed collecting and leaching tanks, in which the top tank is used as a classifier and sand collector and the bottom tank for leaching. The pulp is distributed in the collectors through a Butters and Mein pipe machine or through a hose moved about by a native, who controls classification and collecting, with slat gates and curtains placed at the periphery. The classification is analogous to that of a buddle. Later, the pulp was given a preliminary classification in spitzkasten. The collector is in a closed circuit, the overflow passing to a "safety" spitzkasten, which produces a slime overflow and a sandy underflow returned to the collector. This practice is continued in a few of the old plants. Its weakness is the inability to obtain sufficient



1. Grizzlies
2. Waste sorting belt
3. Rock crusher, jaw or gyratory
4. Dust fan
5. Ore bin
6. Stamps
7. First lift, pump or elevating wheel
8. Pebble bin
9. Tube-mill dewatering cone with diaphragm
10. Tube mill
11. Amalgamation tables
12. Second lift pump
13. Cone classifier
14. Sand/slime cone classifier, hydraulic
15. Sand collection AND treatment tank
16. Slime collector
17. Brown agitators
18. Stock pulper
19. Butters filter
20. Stock solution tank
21. Solution clarifier tank
22. Gold solution tank
23. Precipitation pump
24. Merrill precipitation press
25. Solution sump

FIG. 3.—COMPOSITE FLOW SHEET, 1910 TO 1920. INTRODUCTION OF CONE CLASSIFICATION, SLIME FILTERS AND ZINC-DUST PRECIPITATION. Features: Single-stage sorting and crushing; closed tube-mill circuit; cone classification; sand collecting and treatment in same tank; slime agitation in Brown tanks; Butters slime filtration; Merrill press zinc-dust precipitation.

control. The superimposed tanks were followed by belt transfers of sand between collecting tank and treatment tank, placed in line. Transferring of drained sand was by native labor. Mechanical means were also employed, such as the Blaisdell excavator and Blaisdell distributor. Many of these installations are in current use.

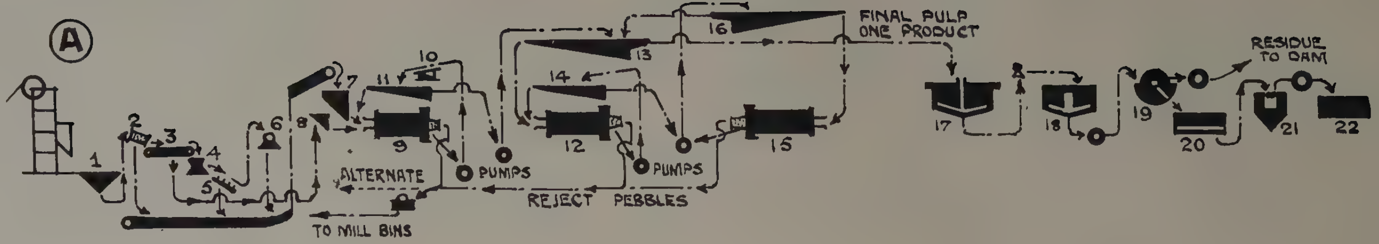
With the introduction of cone classifiers, resulting in clean sand and adjustable slime separation, the practice has been modified in many respects: (1) Collecting and treating in the same tank; (2) dewatering the classified sand on a horizontal continuous filter table (Caldecott table) preceded by a thickening cone and then pumping to the collector with cyanide solution, giving a double treatment, both in the top and bottom tank; or (3) a similar procedure, but using a thickening cone in place of the filter table. System 2 has been discontinued. With the advent of bowl classifiers, a modification of scheme 1 has found favor, and collecting in cyanide solution to increase treatment time.

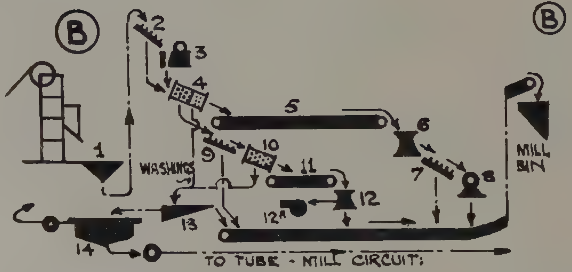
The classified slime was at first stored in dams awaiting a suitable process. This appeared in 1894 in the shape of the decantation process, introduced by Williams, in which successive decantations and washes are carried out in large sloping or cone-bottom tanks. Collecting and settling of slime and decanting of water takes place in the first tank, after which cyanide treatment by decantation is carried out with weak solutions in two or three tanks placed in series, the transfers being effected with centrifugal pumps. The residue is pumped to slime dams, where another decantation is sometimes practiced. As the fine gold in the slime is dissolved rapidly in extremely weak solution, most of the dissolution takes place in the transfer from the collector to the first decanting tank. Later separate and intermediate agitating tanks came into use. These were cone-bottomed tanks with pump circulation, tanks with a Trent agitator, Brown or Pachuca and Dorr air-lift agitators. This process is still being used at some of the old mines where, with a given equipment, its efficiency is governed by the tonnage of feed and rate of settlement. As the first factor is usually in excess of efficient capacity and as settlement is thereby affected and by the temperature and condition of the ore pulp, the process can be considered today to be one of expediency only and one involving considerable gold losses.

After the orthodox decantation process, continuous decantation received attention, also displacement washing in filter presses, the latter being used with efficiency for many years at two plants.

With advent of the vacuum filter (Butters) in 1910 and of the drum-type vacuum filter (Oliver) in 1926 and their demonstration of efficient washing by displacement, decantation was replaced in many old plants, where there were justifiable economic reasons.

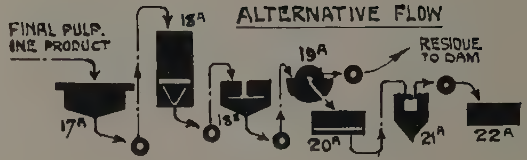
Precipitation of the gold on zinc shavings was part of the MacArthur and Forrest process and has reached a high state of usefulness. It has





B

1. Receiving bin at plant
2. Scalping grizzly
3. Jaw crusher as cracker
4. Trommel
5. Sorting belt
6. Gyratory or jaw crusher
7 Vibrating screen
8. Secondary crusher
9. Vibrating screen
10. Trommel
11. Sorting belt
12. Secondary crusher
13. Rake classifier
14. Thickener
12A. Dust fan



1. Receiving bins at plant
2. Trommel or grizzly
3. Sorting belt
4. Primary crushers, jaw or gyratory
5. Vibrating screen
6. Secondary crusher
7. Mill bin
8. Pebble bin
9. Primary tube mill
10. Corduroy table
11. Dorr rake classifier
12. Secondary tube mill
13. Dorr bowl classifier
14. Dorr rake classifier
15. Tertiary tube mill
16. Dorr bowl classifier
17. Dorr thickener
18. Dorr agitator in series
19. Drum filter or Butters filter
20. Solution clarifier
21. Crowe-Merrill vacuum precipitation system
22. Solution sump

Alternative flow

17A. Collector in charges
18A. Brown agitator in charges
18B. Stock pulper
19A. Drum filter or Butters filter
20A. Solution clarifier
21A. Crowe-Merrill vacuum precipitation system
22A. Solution sump

FIG. 4.—COMPOSITE FLOW SHEET, 1920 TO 1934. STAMPLESS, ONE-PRODUCT PLANT, OR CALLED "ALL-SLIMING."

Features: One-stage or two-stage sorting; two-stage crushing; three-stage grinding in tube mills; continuous cyanide treatment, or alternative charge treatment; drum filtration; vacuum system of zinc-dust precipitation; milling in cyanide solution or water.

not been surpassed by any other method in actual precipitating effectiveness, and is continued in many plants. With initial difficulty of precipitating the gold from the extremely weak solutions in the slime decantation method, large-scale trials were conducted with the Siemens Halske electrolytic method, and several installations resulted. The controversy between the two was finally settled by the improved results obtained with the zinc-lead couple and general improvements in the technique of the shaving process. Clarification of the solution has usually been done through sand, other methods, such as filter presses, tending to choke with lime carbonate and sulfate. Precipitation with zinc dust was established in 1911. The avoidance of gold lock-up and complete monthly returns were attractive. The method became thoroughly reliable with the introduction of de-aeration.

Several composite flow plans (Figs. 1 to 4) have been drawn in which it is endeavored to illustrate the evolution in periods and to guide the reader through the text.

More Recent Current Practice

Current practice presents a great variety of equipment and methods; from old plants of 30 years' standing to the modern examples of the last few years. It is, therefore, proposed to confine this part of the paper to more recent practice. To the uninitiated, the variance in this will be puzzling, but it is explained by a recognition of certain differences in ore characteristics, by local peculiarities and by differences in personal ideas, although the same objective is in view; viz., maximum extraction at minimum cost.

In a general way, the ore in the central and near eastern districts is amenable to simple treatment; at the extreme west, pyrite and pyrrhotite have been more prominent and in one reef the gold is closely encased in the siliceous matrix. In the far eastern districts, the gold is associated to a greater extent with mineral sulfides, and finer grinding is required to bring about contact with the cyanide solvent.

Crushing and Sorting

The improvement in design and types of crushers and the introduction of the stampless milling plant has led to many beneficial changes in crusher station design. More work is now being performed with crushers, and the product leaving this section is much finer, even for the stamp mill. "Choke feeding" of crushers has increased stamp duty by as much as 10 per cent. Mechanical grizzlies and vibrating screens have provided a better division of the run of mine feed for separate sorting of the coarse and fine waste and for two-stage and three-stage crushing. The closed circuit is receiving consideration and is being adopted in at least one plant. Many permutations of equipment are possible. In a few plants, all the run of mine ore is washed and a considerable proportion of the fines screened and dewatered for delivery to the milling circuit. In a chemical sense, this provides an alkaline wash of all the ore prior to contact with cyanide solution. In most cases it is the practice to screen out the fines first and wash only the portion of the rock that is passed to the sorting belts. Pebbles for tube milling are picked off the sorting belts or are separated after sorting by means of grizzlies or trommels. The average waste sorted is 15 per cent of the rock hoisted. In several cases as much as 27 per cent is discarded; the native sorter being trained to pick out waste down to 1½-in. dia. Dust produced by the crushing is withdrawn by means of fans and is laid by means of water sprays, steam or bag filters.

The gradings in Table 2 illustrate final crusher-station product sent to the stamp mill or tube mill direct.

TABLE 2.—*Crusher-station Product*

Plant	+2-in. Square Hole	+1 In.	+¾ In.	+½ In.	−½ In.	Type of Plant
A	nil	12	19	27	42	Feed to tube mills. Stampless plant.
B	nil	3	20	17	60	Composite plant. Feed to tube mills.
B	3	51	24	11	11	Feed to stamps.
C	2	36	14	16	32	Stamps and tubes. Feed to stamps.
D	12	36	20	12	20	Stamps and tubes. Feed to stamps.
E	nil	0.4	5.6	32	62	Stampless plant. Feed to tubes.

Milling

Stamp mills operating today are mostly of the heavy type, from 1750 to 2000 lb. weight, preferably in five-stamp units or the Nissen single unit. Stamping is through coarse screens ranging from 6-mesh per linear inch to 1-in. mesh. Representative stamp duties are from 12 to 33 tons per day. There are some 5650 stamps dropping, the largest single mill being one of 600 stamps. The stamp ratio to tube in the modern plants is as low as 8:1. The different sizes of tube mills in use are 5 ft. 6 in. dia. by 22 ft.; 6 by 16 ft.; 6 ft. 6 in. by 20 ft., 8 by 16 ft. The first two are mostly in conjunction with stamps producing screen products down to 3 mesh. For a coarser feed the larger tubes are favored. Some 416 tubes are in operation. Duties and other data are shown in Table 3.

In the more recent installations mechanical classifiers of the Dorr straight rake type or spiral type are used to serve primary tubes. A mechanized cone classifier (Job machine) is also used in this circuit at one plant. For secondary and tertiary tubes, Dorr bowl classifiers are generally adopted, the bowl having the necessary sensitiveness for the final classification in the "all-sliming" plants. Spiral classifiers are also used in an intermediate position in this circuit. Rake classifiers are usually 8 ft. wide, bowl classifiers 20 to 22 ft. in diameter, spirals up to 15 ft. in diameter.

TABLE 3.—*Duties and Other Data with Pebble Grinding Medium*

Tube Mill	Plant	Load, Tons	Tons −100 Mesh per Day	Tons −200 Mesh per Day	Horsepower	Consumption per Day	
						Balls, Lb.	Pebbles, Tons
		Pebbles[b]					
5 ft. 6 in. by 22 ft. .	C	13–14	157.0	120.0	146		30
5 ft. 6 in. by 22 ft.[a].	B	13–14	120.0	92.0	132		17
6 by 16 ft.[a].	B	13–	140.0	105.0	147		25
6 ft. 6 in. by 20 ft. .	B	15–16	165.0	128.0	235		35
8 by 16 ft.	F	Primary mills		120.5	238		53
		Tertiary mills		231.3	233		28
COMPOSITE GRINDING LOAD OF PEBBLES AND STEEL BALLS							
		Balls[c] Pebbles[c]					
5 ft. 6 in. by 22 ft. .	C	5 11	196	131	160	300	25
6 ft. 6 in. by 20 ft.[d].	A	7 13	253	148	265	500	27
6 ft. 6 in. by 20 ft.[e].	B	6 13.3	271	190	263	420	25

[a] Mine fines have been separated and do not pass to this circuit.

With a demand for increasing tonnage without increasing grinding units, a number of mills are running with a composite grinding load of pebbles and steel balls. The increase in duty is fully 15 per cent and the power increase the same.

Duties of these are shown in the second part of the table.

[b] Ore pebbles fed are 4 to 10-in. mean diameter.

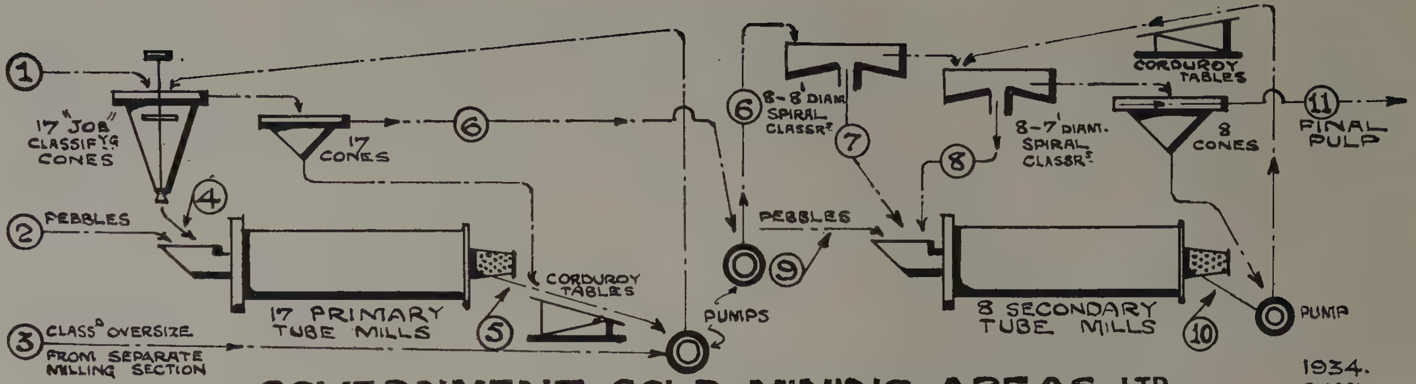
[c] Size of pebbles, 4 to 10 in.; size of balls, 3-in. diameter.

[d] Grinding run of mine ore after crushing.

[e] Grinding screened fines from run of mine ore.

The Job cone classifier is a local invention. It consists of the usual inverted dewatering cone, the underflow valve of which controls the rate of underflow by an ingenious mechanical arrangement.

The spiral classifier is also of local origin, by MacLean. It consists of a cylindrical pan with an upright truncated cone for the bottom. Pulp is introduced at the side in a tangential direction and travels around for about two-thirds of the circumference until it reaches the cut away open third, where it can overflow over a controllable weir. The settled

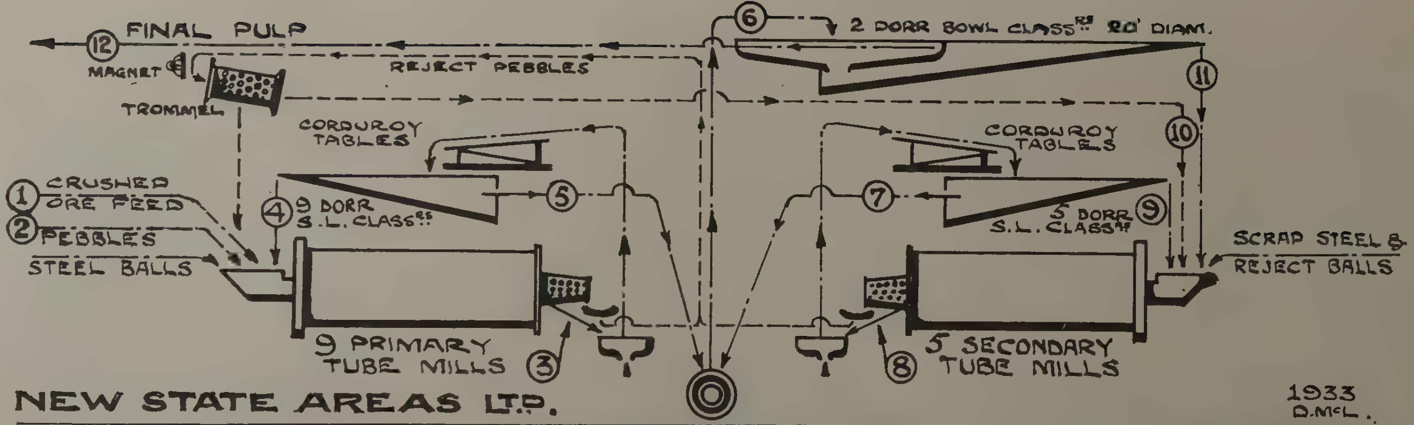


GOVERNMENT GOLD MINING AREAS LTD.

No.	LEGEND.	Tons of Solids per 24 hrs	Specific Gravity	Per Cent Moisture	Ratio L/S	Mesh Analyses. +48	+100	+200	−200
1.	STAMP MILL PRODUCT, −¼" MESH	4107	1·145	79·89	3·97 to 1	66·5	11·5	8·0	14·0
2.	PRIMARY TUBE-MILL PEBBLES	221	–	–	–	–	–	–	–
3.	CLASSD. OVERSIZE FROM SEPARATE MILLING SECTION	2000	1·360	57·96	1·38 to 1	11·0	42·0	28·0	19·0
4.	PRIMARY "JOB" CLASSR. UNDERFLOW PULP	13600	1·970	21·80	0·27 to 1	50·8	37·2	8·4	3·5
5.	PRIMARY TUBE-MILL EFFLUENT	13821	1·825	28·20	0·39 to 1	28·2	40·7	15·4	15·7
6.	PULP FROM PRIMARY TO SECONDARY CIRCUITS	6400	1·190	74·65	2·95 to 1	1·7	26·5	29·8	42·0
7.	8' DIAM. SPIRAL CLASSR. UNDERFLOW	2400	1·710	34·07	0·52 to 1	4·5	56·0	32·8	6·7
8.	7' DIAM. SPIRAL CLASSR. UNDERFLOW	2560	1·710	34·07	0·52 to 1	·3	53·2	40·0	6·5
9.	SECONDARY TUBE-MILL PEBBLES	72	–	–	–	–	–	–	–
10.	SECONDARY TUBE-MILL EFFLUENT	5032	1·710	34·07	0·52 to 1	·6	34·2	34·0	31·2
11.	FINAL PULP	6400	1·095	86·21	6·26 to 1	–	9·5	29·3	61·2

FIG. 5.—TWO-STAGE TUBE-MILL CIRCUIT USING PEBBLE LOADS.

Feed is stamp-mill pulp. Final pulp is classified into sand and slime. Ratio of stamps (1850 lb.) to tubes 200, stamps, 17 primary tubes, 8 secondary tubes. All mine fines are sent to a separate milling section.



No.	LEGEND.	Tons of Solids p. 24 hrs	Specific Gravity.	Per Cent Moisture	Ratio L/S	Mesh Analyses.				
						+1"	+3/4"	+1/2"	+48	−48
1.	CRUSHED ORE FEED	3107	—	—	—	12.0	14.0	26.4	40.6	7.0
2.	PEBBLES TO PRIMARY MILLS	246	—	—	—	+48	+100	+200	−200	
3.	PRIMARY TUBE-MILL EFFLUENT	11410	1.952	22.55	0.29 to 1	36.0	33.9	14.7	15.4	
4.	PRIMARY DORR S.L. CLASSR. RETURN	8104	1.990	20.99	0.26 to 1	49.6	35.8	9.0	5.6	
5.	PRIMARY DORR S.L. CLASSR. OVERFLOW	3306	1.180	75.78	3.13 to 1	3.2	27.9	28.6	40.3	
6.	CIRCUIT PULP	5433	1.172	76.69	3.30 to 1	2.1	16.4	31.4	50.1	
7.	SECONDARY DORR S.L. CLASSR. OVERFLOW.	2127	1.160	78.10	3.56 to 1	—	3.2	37.4	59.4	
8.	SECONDARY TUBE-MILL EFFLUENT	4600	1.775	30.65	0.44 to 1	0.5	15.4	44.6	39.5	
9.	SECONDARY DORR S.L. CLASSR. RETURN	2473	1.920	23.90	0.31 to 1	0.9	30.5	52.4	16.2	
10.	SECONDARY TUBE-MILL PEBBLES	72	—	—	—	—	—	—	—	
11	DORR BOWL CLASSR. RETURN	2080	1.870	26.11	0.35 to 1	5.5	39.6	41.5	13.4	
12.	FINAL PULP	3353	1.115	83.62	5.12 to 1	—	1.9	25.3	72.8	

FIG. 6.—TWO-STAGE TUBE-MILL CIRCUIT USING COMPOSITE LOADS. Feed is crusher product. Final pulp is cyanided as an "all-slime" product.

solids are scraped up the cone incline to the center discharge by a spiral rake revolving in the same direction as the pulp.

Details of conventional grinding circuits are perhaps best described by diagrams (Figs. 5 and 6).

Concentration

The importance of preferential treatment of the sulfide portion of the ore has always been recognized and several methods are employed. In so-called "bulk grinding," use is made of the concentrating action of classifiers in the tube-mill circuit in which sand and much of the pyrite receives preferential grinding. In "selective" grinding an attempt is made to remove the pyrite and give separate grinding.

Concentration in a simple form is practised with corduroy blanket tables in most plants, principally for the purpose of recovering the coarser free gold. Recoveries are from 25 to 60 per cent of the gold content of the ore in a concentrate which, after dressing, is 0.05 per cent of the ore tonnage. The dressed concentrate is sent to amalgam barrels for the recovery of the free metal. Blanketing also provides a safety factor when tube-mill circuits are upset, and it diminishes the "lock-up" of gold in the circuit.

At some plants the Johnson concentrator is used in the tube circuit. It is a rotating drum with a rubber lining, in which riffles or grooves are arranged as in the rifling of a gun. The sulfides collected in the riffles are carried upward, subjected to a washing action and finally discharged at the top of the rotation by means of sprays. This machine recovers approximately 44 per cent of the pyrite and 55 per cent of the gold in a concentrate weighing 8 per cent. After the free gold is recovered by blanketing, the concentrate is subjected to a separate and fine grinding and then combined with the cyanide pulp.

More recently a new type of jig has been developed by Smith and Maxwell at the West Rand Consolidated mine. It is of a type in which the screen frame and its contained bedding is given a jigging movement. This frame is suspended at the top of a large cone some 8 ft. in diameter, to which it is joined by a flexible rubber joint fastened to the cone circumferentially. The movement is imparted by an overhead mechanism. The feed is at the center and the tailings overflow around the circumference. A spigot concentrate product is discharged at the cone apex. This jig is placed in the closed tube-mill circuit one to each tube. The concentrate from the rougher machines, after dressing with cleaning jigs and James tables, constitutes about 2 per cent of the ore weight. It is ground in ball mills and cyanided separately, after which it is delivered to chemical works for sulfuric acid manufacture. Free gold is recovered by means of blankets placed in the cleaning and grinding circuit. This machine has a large capacity.

Experimentation with flotation is proceeding and the possibility of the use of this metallurgical tool in the flow plan is receiving attention.

Cyanidation

In cyanidation there has been a pull away from sand leaching toward one-product agitation treatment, induced by the improved extraction with finer grinding, lower capital cost and simplicity. All new plants, erected during the past 10 years, have been on this order. Such a plant, however, cannot be stretched readily as regards capacity. Even in the older plants, extensions have been preferably made to the grinding and sliming section, so that the ratio of sand to slime treatment has become as low as 10:90, and 20:80 in some instances, from a former ratio of 60:40. Leaching, in general, is carried on in the orthodox way with solution: solid ratio of from 1:1 to 2:1 and the treatment time of three days to seven days. In one case, on high-grade sand and ample tank room, treatment is extended to about 20 days. Maximum solution strength is about 0.06 per cent KCN with an alkalinity of 0.005 per cent CaO. Solutions usually are added over several periods with draining between. A water wash is seldom practicable. Aeration of the lixiviating solution, recently revived by Prentice, is receiving attention on most plants in various forms.

During the current year, a modification has been adopted at one plant, the feature of which is the introduction of a small volume of air under pressure during the first two periods, and drainage assisted by vacuum during the last two or three periods. The result has been a more thorough oxidation of ferrous compounds, a lessening of cyanide consumption and shortened treatment. The method is explained in Fig. 7. The air is forced from below the filter mat for some 5 hr. during the first and second saturation periods. This differs from Homestake practice, where the air is admitted while the sand tank is in a drained condition. The application of a vacuum to the drainage is a revival of early practice, but with modern and large dry vacuum pumps.

In slime or one-product pulp treatment, agitation is effected by Brown tanks or Dorr agitators, either in charges or in series. According to the value and fine condition of the pulp, solution strengths for this are from 0.01 to 0.025 per cent KCN, and agitation from 6 to 30 hr. More attention is being paid to an even and adequate air supply and some form of pre-aeration. Brown tanks in use are 15 ft. in diameter by 45 ft. deep, holding a 100-ton charge; others of 22 by 45 ft. holding 250 tons, and a few 33 by 45 ft. holding 500 tons. The liquid solid ratio is usually 1.20:1.

With the exception of the decantation plants, the dissolved gold is recovered by displacement washing in Butters filters or continuous drum Oliver filters, or that type. Most new installations employ the drum filter (16 ft. diameter by 14-ft. drum) as fitting in with the continuous system and being subject to closer observation. High duties are obtained on the permeable siliceous material with these filters, 4.5 tons per leaf per day with Butters and 0.65 tons per day per square foot with Olivers being common. A single stage only is used. A second stage has not been found economical. Washing displacement of dissolved gold is excellent, the difference between total and washed residue being 0.005 to 0.02 dwt., according to wash permissible by filter duty. Cyanide consumption ranges from 0.15 to 0.50 lb. per ton treated, the higher

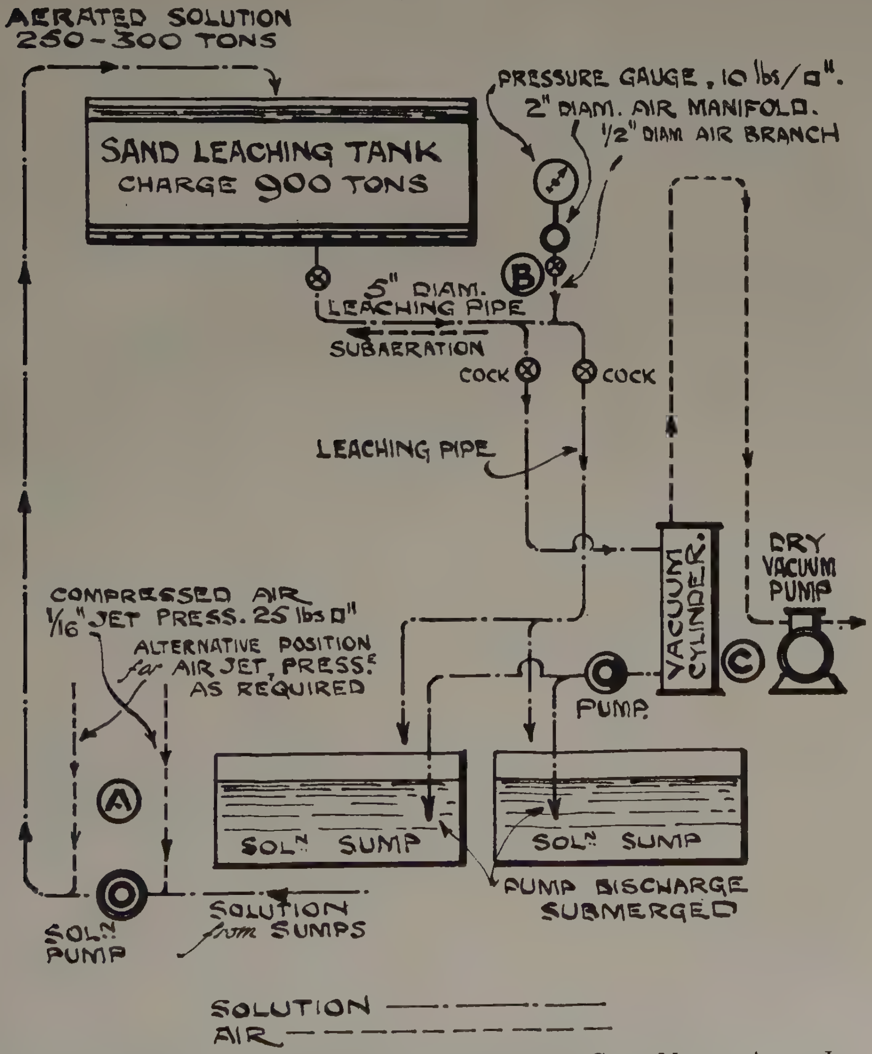


FIG. 7.—DIAGRAM OF SAND TREATMENT, GOVERNMENT GOLD MINING AREAS LTD. AERATION OF SOLUTION, SUBAERATION AND VACUUM DRAINAGE, JUNE 1, 1934.

tendency being on plants grinding in solution and having a large surface area of solution exposed.

Precipitation of the gold and the silver from the solutions is effected by both the older zinc-shaving system and by zinc dust; all new plants, however, are using the dust and the Crowe-Merrill de-aeration and vacuum leaf apparatus. De-aeration of solution in preparation for precipitation is found to be advantageous with many of the zinc-shaving installations, where this has eliminated the hydrated zinc oxide (white precipitate). With decantation, slime treatment and the weak cyanide solution used (solutions going to precipitation as low as 0.0035 per cent KCN strength) the shaving process with the zinc-lead couple has held its own and therefore many older plants have found the older method more suitable. Zinc dust is used on the stronger solutions delivered from one-product treatment and on the stronger mixed solutions from sand-slime treatment where Brown tanks or other thick-slime pulp agitators are used. The two methods today divide precipitation about equally between them (Table 4). Their controlling characteristics may be summed up as follows:

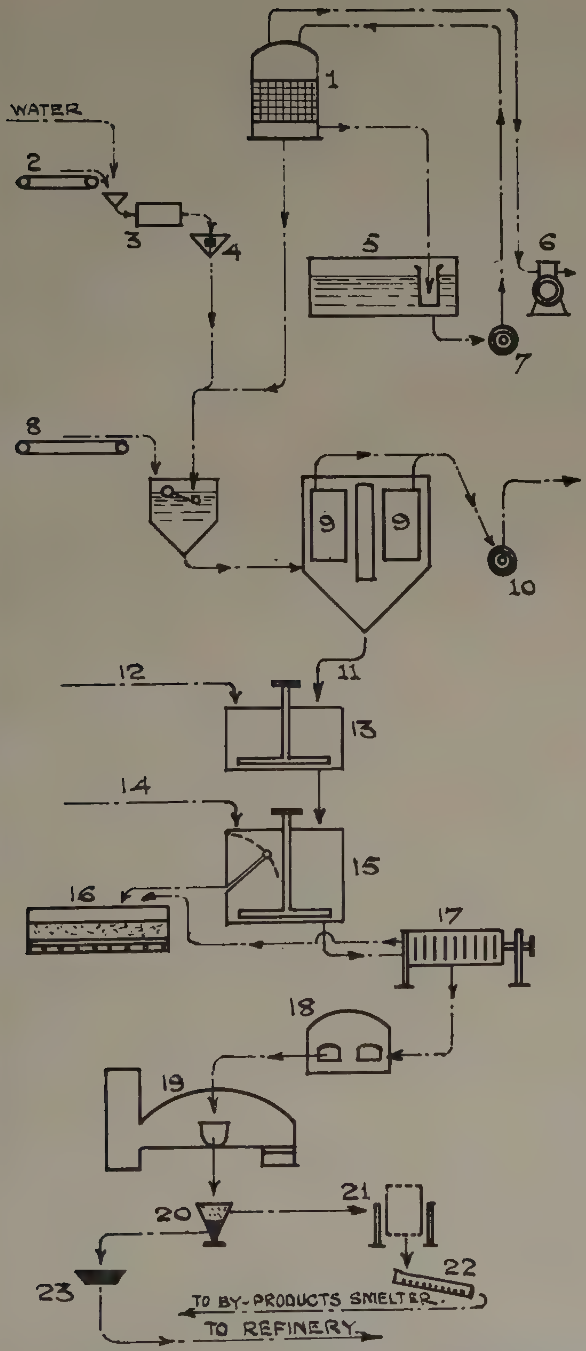
Zinc Shaving.—Efficient precipitation of both strong and extremely low cyanide strength solutions, of low protective alkalinity; requiring comparatively little technical supervision; large space required; laborious and sloppy clean-up; fluctuating and at times large gold lock-up.

Zinc Dust.—Efficient precipitation if minimum cyanide strength is higher than with shavings; high alkalinity; close technical supervision; small space required; convenient and rapid clean-up; no gold lock-up; lower zinc consumption; lower cost with precipitate treatment included.

TABLE 4.—*Technical Data of Precipitation*

Method	Precipitated Solution Assay, Dwt. per Ton	Solution to Precipitation, Per Cent		Zinc Consumption, Lb. per Ton Milled	Solution Precipitated per Ton Treated, Tons
		KCN	CaO		
With shavings					
Sand solution	0.02	0.028	0.005	0.14	1.3
Slime solution	0.015	0.011	0.005		2.0
With dust					
Mixed	0.015	0.016	0.02	0.05	1.5
From one-product pulp	0.020	0.014	0.018	0.06	1.8

The zinc-gold precipitate or "slime" is treated with acid, calcined and smelted in large (No. 100) clay-lined graphite pots in reverberatory furnaces holding 25 to 32 pots, or it is smelted in reverberatory furnaces to lead bullion and cupelled. Both methods are about equal in cost. The lead smelting locally called the Tavener process requires greater



1. Deaeration vacuum cylinder and filming lattice
2. Lead salt feeder
3. Ball mill emulsifier
4. Cone feed
5. Solution sump and overflow seal
6. Vacuum pump, 22-in. vac.
7. Solution pump
8. Zinc-dust feeder
9. Precipitate filter leaves
10. Precipitated solution pump
11. Zinc-gold slime
12. Sulfuric acid
13. Acid tub for dissolving base metals, etc.
14. Hot water wash
15. Washing and decanting tub
16. Sand filter
17. Filter press
18. Calcine furnace
19. Reverberatory pot furnace
20. Conical molds
21. Slag to Krupp ball mill
22. Shaking riffle
23. Buttons melted into bars

FIG. 8.—PRECIPITATION BY ZINC DUST (CROWE-MERRILL PROCESS) AND TREATMENT OF PRECIPITATE, JUNE 1, 1934.

skill and there is an absorption of gold in the furnace hearth. Short cuts in the elimination of acid treatment or direct smelting have not proved satisfactory. The zinc sulfate byproduct is used for underground timber treatment. Lead salt, nitrate or acetate is used in all precipitation. With zinc dust it is added to the solutions before precipitation in an amount of approximately 5 lb. per 1000 tons of solution. At one plant, after considerable research and experimentation, the scheme outlined in Fig. 8, which also shows the precipitation plant, has been adopted.

The bar gold is sent to the central refinery, where refining is carried to a 996 gold and 998 silver fineness by the chlorine process. Byproducts from the reduction works are lead smelted at a central smelting works, which produces gold bullion, coppery matte and speiss. In the speiss there is a concentration of platinum group metals, particularly osmiridium.

Chemical

On the chemical side, current practice has the advantage of the basic work carried out during the establishment of the cyanide process. Although the field for investigation has been narrowed, there is progress. The subject of the neutralization of acid ore underground, the chemistry connected with sand-filling old workings and the neutralization of underground waters, which form a large portion of the supply needed for ore treatment, receive attention and are controlled. Hydrated lime is used in large quantities underground and again on the surface, to render this water suitable. In special cases of waters near $CaSO_4$ saturation, carbonate of soda is added to prevent undue scaling. Unslaked lime, to the extent of approximately 2 lb. per ton of ore, is used in cyaniding, a certain amount being added during milling and the remainder during cyanide treatment. Oxygen content of solutions, both for de-aeration prior to precipitation and for aeration of dissolving solutions, is receiving close attention and study. Information as to the interplay of oxidizing reactions within the sand charge is obtained by following ferrocyanide content of leaching solutions. The addition of lead salts, except in precipitation, is not common. Oxidizers, like permanganate or bleaching powder, are applied only in special cases and principally to oxidize organic matter in the water supply or in accumulated tailing or underground sludge. The cyanide used is sodium cyanide in strength from 48 to 96 per cent. Several metallurgical and chemical laboratories serve the industry.

Tonnages, Recoveries and Extraction

The scale of ore treatment is shown by the tonnages and recoveries for the month of May, 1934 (Table 5). Of these, Crown mines, of Central mining group, holds first place in production with 86,242 oz.; Randfontein Estates Gold Mining Co., of Barnato group, is first in

TABLE 5.—*Tonnages and Recoveries during May*, 1934

Groups and Individual Mines	Tons Milled	Ounces Gold Recovered	Number of Stamps	Number of Tube Mills
Central mining group	1,316,600	312,219	2,698	146
Barnato group	885,000	230,383	1,230	104
Anglo-American group	385,000	111,553	225	59
New Consolidated Gold Fields group	234,200	81,268	560	28
Union Corporation group	227,000	72,709	210	40
Albu group	180,000	37,686	250	23
New Kleinfontein mine	55,100	9,473	190	4
Witwatersrand Deep mine	52,000	10,331	170	6
Luipaardsvlei mine	42,000	9,282	70	6
	3,376,900	874,904	5,603	416

TABLE 6.—*Extractions*

Plant	Type of Plant	Percentage Grinding to		Total Extraction (Recovery + Residue), Per Cent	Head Value, Dwt.	Residue Value, Dwt.
		−100	−200			
B	Sand and slime	92	66	96.73	7.69	0.251
D	Sand and slime	78	58	96.35	4.91	0.179
G	Sand and slime	88	63	96.85	4.15	0.131
A	One product	97	76	96.72	8.52	0.280
F	One product		89	96.37	7.29	0.265
H	Sand and slime	78	57	97.1	6.87	0.197
I	Sand and slime	80	49	95.6	4.10	0.182
J	Sand and slime	83	60	96.0	5.09	0.199

TABLE 7.—*Representative Gradings*

Plant	Pulps and Products	Percentages				Per Cent of Tonnages
		+48 Mesh	−48 +100 Mesh	−100 +200 Mesh	−200 Mesh	
B	Final pulp to cyanide works		8	26	66	100
	Sand		26	56	18	26
	Slime		1	16	83	74
A	One-product final pulp		3	21	76	100
F	One-product final pulp				89	100
D	Final pulp to cyanide works	1	21	20	58	100
	Sand	2	61	29	8	68
	Slime		4	15	81	32

tonnage, with a single plant, having 600 stamps and 24 tube mills, and milling 330,000 tons for the month. No direct comparison can be made from these stamp and tube data because weights of stamps, size of tubes and degree of grinding are too greatly at variance. Extractions are shown in Table 6 (all plants with vacuum filters).

Gradings.—This simple subject is important, particularly where extraction is dependent on degree of comminution and where large-scale milling technique must be largely governed by a measurement of particle size. Representative gradings of various pulps and classified products are given in Table 7.

TABLE 8.—*Costs in Shillings per Ton Milled*

Monthly Tons Milled	Transport Shaft to Plant	Crushing and Sorting	Stamp Milling	Tube Milling	Cyaniding	Total
79,000[a]	0.040	0.509	0.565	0.645	0.911	2.670
208,000[b]	0.190	0.425	0.441	0.914	0.774	2.744

[a] Plant grinds to 77 per cent −100 mesh and sorts 27 per cent waste.

[b] Plant grinds to 92 per cent −100 mesh and sorts 23 per cent waste. This is a combination plant where all mine fines pass directly to tube mills.

TABLE 9.—*Power Consumption*

KILOWATT-HOURS PER TON MILLED (INCLUDING CLASSIFIERS AND AUXILIARY MACHINES)

Plant	Crushers		Stamps		Tube Mills		All Pumps in Milling		Cyanidation		Total
	Kw-hr.	Per Cent	Kw-hr.	Per Cent	Kw-hr.	Per Cent	Kw-hr.	Per Cent	Kw-hr.	Per Cent	Kw-hr.
a	0.884	3.51	nil	nil	18.58	73.69	1.25	4.96	4.49	17.81	25.204
b	0.824	3.60	2.77	12.11	13.023	56.94	2.99	13.07	3.26	14.25	22.87
c	1.062	5.19	3.291	16.10	11.421	55.86	1.541	7.54	3.132	15.27	20.447

KILOWATT-HOURS PER TON −100 AND −200-MESH PRODUCED. CRUSHING AND MILLING (EXCLUDING PUMPS; INCLUDING CLASSIFIERS)

	Stamps		Tubes		Stamps and Tubes		Stamps, Tubes and Crushers	
	−100	−200	−100	−200	−100	−200	−100	−200
a	nil	nil	19.095	24.383			20.004	25.543
b	27.361	42.379[d]	15.884	22.407	17.146	24.427	18.040	25.702
c	19.145	28.717	18.211	26.473	18.412	26.944	19.741	28.889

[a] Plant is all-sliming.

[b] Plant is composite with stamps and tubes and sand-slime treatment.

[c] Plant is orthodox stamp-tube, sand-slime plant with rather hard ore.

[d] Only coarser rock fed to stamps.

Costs

The cost of ore reduction from shaft head to residue dump is influenced largely by the layout and age of the plant, amount of sorting and degree of comminution, assuming like tonnage conditions. The examples of Table 8 are illustrative.

Power

With few exceptions, power is electric from central stations. A few interesting data on consumption for crushing, milling and cyanidation are given in Table 9.

General Design and Mechanical Features

With the gently rolling nature of the Witwatersrand topography, the reduction plants are built on practically level ground or with a fall of about 4 per cent. Heavy slopes do not lend themselves to the return circuits and to the final disposal of residues. The present plant is under cover with the exception of cyanidation tanks, sumps and steady heads. Centrifugal pumps are in general use for pulp elevation and transport, have been designed in large sizes (to 14 in.) and are extremely reliable. Conveyor belts are used for rock transport to bins and in intermediate stages; also for sand transfers. With the employment of the Kaffir as the ordinary laborer, simplicity in detail has been aimed at; intensive mechanization is of less importance.

As all residues must be conserved, the landscape is dotted with sand dumps and slime dams. The former is usually served with endless rope haulage and the latter by pump with a return of settled water or solution.

Organization

Most of the mines come under the control of groups or houses, being administered as regards general policy, both in financial and technical matters, by the group head office staffs. The mines are members of the Chamber of Mines, which is instrumental in coordinating such matters as white labor, native labor, central smelting works and refinery, and subjects that concern the general welfare of the industry. By such organization and with a noncompetitive industry, which permits an interchange of information, isolation of individual mines is avoided, the benefit of collective experience and knowledge becomes available, and action may be concerted.

It may be remarked that although nature has laid down a comparatively simple ore, uniformity in treatment has not been achieved. Such a state is not likely to be reached, as the science of ore reduction, like other sciences, is dynamic and never reaches an end point. Development will not necessarily march on an even front.

ACKNOWLEDGMENT

It has been necessary in this paper to draw on the knowledge of colleagues and their work, recorded in technical publications. Grateful acknowledgment is made here. Authorities are not specifically quoted, but may be found in the appended bibliography.

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13. H. A. White: Crushing and Grinding of Banket. *Jnl.* Chem., Met. and Min. Soc. South Africa (July, 1929).
14. H. A. White: Cascading and Cataracting in Tube Mills. *Jnl.* Chem., Met. and Min. Soc. South Africa (July, 1930).
15. F. Wartenweiler: The Use of Composite Tube Mill Loads. *Jnl.* Chem., Met. and Min. Soc. South Africa (May, 1932).
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17. Willey and Ewing: Stage Tube Milling and Selective Grinding at West Springs. *Jnl.* Chem., Met. and Min. Soc. South Africa (October, 1930).
18. H. A. White: Absorption of Gold in Tube Mills. *Jnl.* Chem., Met. and Min. Soc. South Africa (December, 1930).
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20. E. H. Johnson: Johnson Concentrator. *Jnl.* Chem., Met. and Min. Soc. South Africa (April, 1927).
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Mechanical Preparation of Nonmetallic Minerals

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The term "milling" as applied to nonmetallic minerals often refers merely to pulverizing without preliminary beneficiation. As applied to dimension stone, it embraces all the steps involved in shaping and giving the desired finish to blocks. Processes such as sawing, planing, rubbing and polishing seem far removed from milling as the term is used in ore dressing, nevertheless they involve the element of concentration, for only 25 to 50 per cent of the gross quarry output may leave the mill as finished products after imperfect stone has been culled and the rough blocks trimmed. On the other hand, numerous nonmetallic minerals have to be concentrated in much the same manner as low-grade ores. Engineers and machinery manufacturers have begun to recognize more clearly the opportunities awaiting them in the nonmetallic field and have brought with them the methods and equipment developed in metal-mining camps, occasionally with little modification.

General Economic Considerations

Approximately one-half of the total value of the mineral production of the United States represents mineral fuels, and the other half is about equally divided between metals and nonmetals, the nonmetals recently having become relatively more important. In terms of volume the domestic output of nonmetallic minerals other than fuels is more than 10 times the annual output of metals. Considered on the basis of industrial utility and general commercial and military significance, moreover, the flow of nonmetallic mineral products is quite as needful to our national integrity and well-being as is that of metals, fuels, or other basic materials.

Despite their importance individually and as a group, the nonmetallic mineral industries differ so widely in essential characters and economic environment that producers thereof have been slow to develop a community of interest among themselves or with the metal-mining fraternity. A spirit of change is in the air, rule-of-thumb methods soon may be relegated to limbo, and new methods—especially time-tested ideas borrowed from ore-dressing practice—are being introduced. A faithful record of plant practice in certain of these fields, however, must register

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methods, equipment, and even habits of thought that on first blush seem antiquated or inefficient to engineers schooled in metal-mining operations. The present paper essays to hold the mirror to present-day technology, revealing trends only insofar as they are reflected in commercial applications and without venturing rashly to recommend what ought to be done. In fact, before submitting outlines of current practice in individual industries, it seems proper to point out that there are fundamental economic considerations in these industries as a group that apply to the production of ores and metals to a smaller degree or not at all. For example:

1. The marketability of most nonmetals depends upon inherent physical as well as chemical characteristics. In selling copper concentrates differences in color or even in grain size are of minor consequence, but for dimension stone or a pigment they may be of paramount importance.

2. The field of beneficiation may be narrowly limited. To mention only a few examples: a marble slab that contains a natural flaw or that is damaged in milling cannot be melted and recast; off-color clays or iron-bearing glass sand ordinarily cannot be purified except at prohibitive cost; short-fiber asbestos cannot be spliced for spinning purposes; no amount of processing will convert a black diamond to a first-water gem; graphite is valued according to the size and nature of the flake; and the potash-soda ratio of feldspar can be modified only by blending or preliminary hand sorting.

3. Consumer acceptance for a new product is impeded by intrenched buying habits which in turn are due in part to lack of known standards. Often the suitability of a given material is specified merely according to its present source of supply, and a proposed substitute must be tried out in a practical plant test, and even new formulas may have to be worked out before it can be employed successfully. In addition to technical manufacturing problems, questions of style and even hard-shell prejudice may be encountered.

4. Lack of a recognized market, such as is afforded for ores by the large smelting establishments, renders preliminary estimates of prices more or less uncertain, as in the last analysis the realized value may depend upon private negotiation between buyer and seller.

5. Producers of nonmetals are generally required not only to mine their material but also to carry the processing to the point where it can be sold as a relatively finished product. Whereas the smelters act as shock absorbers between the ore miner and the ultimate consumer and relieve him of the responsibility and capital expense incident to smelting and refining, the nonmetallic producer generally has to undertake the manifold risks of an integrated enterprise.

6. The life of a plant is determined not only by the size of the deposit but by the life of the market, often strictly a local one. Transportation

frequently constitutes a relatively large percentage of the delivered cost, and since freight or trucking may even exceed plant cost per ton on such items as sand and gravel, portable plants may be more economical than better equipped permanent installations.

ADDITIONAL REFERENCES AND ACKNOWLEDGMENTS

Within the space alloted for this paper it has been impossible to indicate in more than barest outline the various means employed for preparing the different nonmetallics for market. For somewhat more detailed information, the reader is referred to Ladoo's compendium[1] which has been freely consulted in preparing the present review. With respect to individual items, the footnote references are appended not merely in acknowledgment but also as guideposts to more detailed information or typical flow sheets. For additional references to plant practice there is no better single source than the lists presented by Robert H. Richards and Charles E. Locke in their reviews of progress in ore dressing and coal washing published in the annual volumes of Mineral Industry. On a growing number of specific commodities the publications issued by the United States Bureau of Mines and by the Canadian Department of Mines should be consulted. Useful data may also be obtained from mannfacturers of certain equipment; in fact, some of the material utilized in the present paper was suggested or amplified by perusal of advertising literature of such firms as the Dorr Co., the Hardinge Co., Raymond Bros. Impact Pulverizer Co., Robinson Manufacturing Co, Sturtevant Mill Co., and the Whiting Corporation.

My deepest obligation is to my teacher and friend, Prof. Charles E. Locke, for lifelong inspiration and for wise and unfailing counsel. Grateful acknowledgment is also made to W. H. Coghill, Oliver Bowles, A. H. Emery, Hewitt Wilson, B. W. Gandrud, and others of my colleagues in the Bureau of Mines, as well as to many friends and acquaintances elsewhere.

ABRASIVES[2]

Natural and artificial abrasives both require careful mechanical preparation before they can be marketed. A special feature is the necessity of retaining sharp edges on the grains. Millstones, buhrstones, pulpstones, chaser stones, grindstones, whetstones and tube-mill linings, made from suitable selected siliceous rock, are roughly trimmed at the quarries and subsequently dressed to size. For the manufacturer of bonded grinding wheels, natural abrasive grains have been largely superseded by silicon carbide and artificial corundum, but even these must be crushed and carefully sized; in fact, grinding wheels are graded by fineness of grain ("grit") as well as hardness ("grade") or degree of bonding. Sandpaper and other coated abrasives likewise have to be closely sized. *Silicon carbide*, after being ground to 8 or 10 mesh in a ball mill, is agitated with steam and water to float off graphite; further agitated first with caustic soda (to dissolve silicon and silicides and eliminate the last of the graphite), second in neutralized solution (with H_2SO_4), and third in hydrochloric acid solution (to dissolve iron); and finally washed in a screw washer. Overflow is elutriated for flour grades, and sand sizes are dried, passed over a magnetic separator, and screened. *Fused alumina* is broken by a falling weight and then sledged to 12-in. blocks, which are reduced by graded crushing in jaw crushers and rolls to 6 mesh. A magnetic separator removes magnetic iron, and the nonmagnetics are roasted to render the remaining iron magnetic, and deslimed in Dorr equipment, the overflow from which is settled, dried and bagged for market. The sands are dried, given another magnetic treatment, and then graded into about 24 sizes in the same manner as silicon carbide and other grains. Silk bolting cloth has been used almost exclusively for grading finer sizes of all abrasives, but wire cloth has been improved as to uniformity and screening capacity. Products from each screen must be checked continually by small hand sieves. For lithograph work and certain other trades requiring the highest accuracy of grading, the various products are resifted through "proving silk."

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[1] R. B. Ladoo: Non-Metallic Minerals. Occurrence—Preparation—Utilization. New York, 1925. McGraw-Hill Book Co.

[2] V. L. Eardley-Wilmot: Abrasives (4 vols.). Canada Dept. Mines, Mines Branch, Ottawa, 1927.

W. M. Myers and C. O. Anderson: Garnet: Its Mining, Milling, and Utilization. U.S. Bur. Mines *Bull.* 256 (1925).

U.S. Dept. of Commerce. Simplified Practice Bull. R118.

Pumicite, *tripoli* or *rottenstone* are pulverized dry in hammer mills, sometimes followed by wet grinding in tube mills, the product being treated in wet classifiers or by an intricate system of driers, pulverizers, screens and air separators.

Grecian (Naxos) and Turkish *emery* is shipped to the United States in hand-selected lumps, ranging from about ½-in. size up to 25-lb. fragments. It is crushed in jaw crushers, a series of rolls, and finally in a ball mill. Mica may be simply blown out as the coarse grain drops down a chute, but in some plants it is washed in a sort of chaser mill or Chilean mill with hardwood wheels. *Corundum*, on the other hand, generally needs a preliminary concentration by gravity methods. In South Africa, as well as in Canada and the United States, jigs and tables have been employed, but much of the South African crude corundum is cleaned by crude methods of hand screening and washing alluvial material. Magnetic separators may be necessary to remove iron minerals.

Most of the domestic production of abrasive *garnet* has come from Warren County, New York. Pink (almandite) garnet occurs in crystals or nodules up to several inches in diameter, comprising up to 13 per cent of a gneiss. Hornblende, which has almost the same specific gravity as the garnet, is the principal gangue mineral, along with feldspar and a little pyroxene and mica, quartz, and iron sulfides. The main objective is to avoid fines, as the abrasive paper trade is the principal outlet for the material. For plate-glass polishing, however, all sizes are finally ground to under 300 mesh. Stage crushing is the rule, and as garnet does not break down easily the undersize from a fine screen may often be discarded. Harz or James jigs are commonly used for concentrating all sizes over about 14 mesh. Sands may be treated on tables, but the Hooper vanning jig (which was developed at a New York garnet mill) preceded by a classifier apparently accomplishes at least as good results, although concentrates have to be removed by hand. Dry concentrators have been used successfully for garnet concentration in New Hampshire and North Carolina, where the gangue consists mainly of mica and feldspar or quartz. Electrostatic cleaning is an effective means of eliminating mica from concentrates, but a second tabling of the carefully sized product is also effective.

AMBER[3]

The blue-earth deposits of East Prussia and contiguous territory in Lithuania, from which most of the world's amber is obtained, lie along the coast and extend far out under the Baltic Sea. The nearly horizontal bed of blue earth lies about 130 ft. beneath the surface of the land but is uncovered on the floor of the sea several hundred yards from shore. Pieces of clean amber are washed ashore from time to time, and formerly production was confined to beachcombers, fishermen, and deep-sea divers, but at present probably 90 per cent of the output results from open-cut mining operations, the blue earth bearing the amber being taken up an incline to the surface in 7-yd. cars and transported to Palmnicken, where it is dumped on a grizzly and washed down by hydraulic nozzles. The larger pieces of amber are removed by hand, and the smaller ones are washed away with the clay slurry. Because of its low specific gravity (under 1.5) the amber remains in suspension, while the heavy material is removed (like slate in coal washing). It is recovered from the amber-bearing slurry and sized in a classifying drum or trommel. The larger pieces are cleaned of their crust in special wooden drums to which sand and soda solution are added and undergo a further cleaning during the final sorting at Königsberg, where they are beaten by hand so that only clean amber will be sent to market. In Burma hand sorting only is employed, the debris hoisted from the primitive deep-mining operations being hoed over by a coolie before being sent to the dump.

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[3] F. Prockat: Amber Mining in Germany. *Eng. & Min. Jnl.* (1932) **129**, 305–307.

L. L. Fermor: General Report of the Geological Society of India for the Year 1930. *Rec.* Geol. Survey of India (1931) **65**, pt. 1, 33–34.

ASBESTOS[4]

Inasmuch as the value of a given quality of asbestos depends mainly upon the length of fiber, the general principle of removing the finished product and rejecting the waste from the mill circuit as soon as possible applies with double force. Fiber once freed from rock should never be allowed to enter the next crushing unit, and excess sand also tends unduly to cut up fiber with which it may come in contact. As exemplified by Canadian practice, asbestos recovery, though varying in detail, embodies essentially the following sequence of operations: (1) Coarse crushing, generally in 36 by 24-in. jaw crushers set with 4 or 6-in. discharge opening; (2) picking belt, where crude fiber, wood fragments, and tramp iron are removed (sometimes discharge from first belt is tumbled upon a second belt directly or after intermediate crushing); (3) drying (rotary or stack driers); (4) recrushing in stages, at the same time opening up or fluffing the fiber, each successive crushing being followed by screening out fine sand and lifting the freed and open fiber by air suction to collectors; (5) cleaning and grading; and (6) bagging for shipment.

Asbestos-bearing rock is preferably reduced by straight impact and disintegration. A minimum of grinding or shearing is desired, and the fiber needs not only to be freed from the rock but also to be "fluffed" or opened up. The first mill breaking of rock may be done in a jaw or gyratory crusher, hammer mill, or rolls, but subsequent crushing or "fiberizing" is done in cyclones, of which the locally built "Jumbo" is generally accepted as superior. A Jumbo consists of a cylindrical shell lined with suitable plates within which revolves a horizontal shaft fitted with a series of arms carrying "beaters." The rock, fed from above at one end, is made to pass through the machine. The beaters are made in two parts, a permanent arm bolted to the shaft and a manganese-steel hammer bolted to the end of the arm. The arms and hammers are inclined with respect to the shaft so as to direct the rock forward from the feed end to discharge. The length of a Jumbo is 6 or 8 ft.; the interior diameter is 24 or 36 in.; beaters are set at intervals of 6 or 8 in. along the shaft and clear the inside of the shell by about ½ to 1 in. The shaft is revolved at 400 to 800 r.p.m., according to the nature of rock treated. In the Laurie cyclone there are two cast-steel impellers shaped like a ship's screw, which revolve in opposite directions at a speed of 1700 or 2000 r.p.m. The Pharo cyclone is similar except that the impellers revolve in the same direction. The Torrey cyclone is somewhat like the Jumbo except that it is a vertical instead of a horizontal machine.

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[4] J. G. Ross: Chrysotile Asbestos in Canada. Canada Dept. Mines, Mines Branch, *Bull.* 707 (1931) 37–56.

W. A. RuKeyser: Asbestos Mining and Milling in Quebec. *Eng. & Min. Jnl.* (1922) **113**, 617–25, 670–77.

W. A. RuKeyser: Asbestos Milling in Quebec. *Eng. & Min. Jnl.* (1932) **133**, 102–106.

W. A. RuKeyser: Asbestos Milling in the Urals. *Eng. & Min. Jnl.* (1933) **134**, 415–419.

From each crusher, hammer mill, or fiberizer, the broken rock falls on, or is conveyed to, a screen where the sand is shaken out, the fiber lifted by air suction at the lower end, and the oversize passed to the next crusher. The lifted fiber is collected in conical steel tanks, sometimes being pulled through the fan blades to assist in shaking out dust. The dust-laden air is settled and filtered; the "floats" are bagged and sometimes are graded by screening into two products. From the collectors the mill fiber is dropped to the graders, which are a series of flat or rotary screens. The final grading is generally done in slowly revolving hexagonal or octagonal screens equipped with rotating paddles to push the fiber through the screen cloth. Each graded product is generally recleaned by screening or air floating.

In the Thetford and Black Lake operations in Quebec, a yield of 3 to 10 per cent of fiber is effected in most mills, occasionally as high as 15 per cent. In Broughton (slip-fiber quarries) extractions range from 7 to 20 per cent. Operating costs naturally vary widely, probably between $10 and $60 per ton of fiber (including mining). In 1929, the average yield from all Quebec quarries was 99.76 lb. of asbestos valued at $2.215 per ton of rock mined, compared with 103.2 lb. valued at $2.11 in 1928. Only a little over 20 per cent of the crude rock hoisted is sent to the waste dump without milling, so it is evident that milling costs have to be low; probably not over $1 a ton and considerably lower in many of the larger mills. In Vermont the yield (all short or nonspinning fiber) is around 6 per cent.

BARITE[5]

Domestic output, mainly from Missouri, Georgia and Tennessee, is mostly from residual deposits wherein the barite occurs as nodules in clay. Standard practice involves log washing to remove the clay, followed by hand sorting and jigging and perhaps table treatment of the fines. Despite the low value of the product, generally under $8 a ton, the ratio of concentration may be as high as 20 to 1, but the product leaving the logs after removing the clay generally yields at least 25 and often over 50 per cent of barite. Temporary type of construction is the rule; many plants are not covered except for a shed over the picking belt, jigs and motors. Although poor design obviously is inexcusable, due consideration should be given when building a new plant to the low value of the product, availability of local cheap labor, and the fact that 3 or 4 years at most represents the useful life of a deposit, after which the equipment may be moved to a new site. At small pits much of the output is from hand operations, the barite being freed from clay and other impurities with small hatchets; after drying it may be further cleaned by vigorous shaking in a "rattler," a box with a screen bottom through which clay and sand are sifted out, leaving only lump material. In certain vein deposits, as in California and Nevada, no concentration is needed; the ore is loaded on cars just as it comes from the mine with only a small amount of hand sorting. However, at one California plant employing Woodbury jigs and making a wet-ground product (sent to cone classifiers and splash drier), additional concentration was obtained in the Hardinge mill used for grinding. By adding only an equal volume of water, the pulp density was kept around 3, thereby floating quartz particles out of the mill so that they could be eliminated by a short screen at the discharge end. In Great Britain, as well as in the United States, a small amount of barite may be produced as a middling or byproduct from tabling lead, zinc or fluorspar ores.

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[5] W. M. Weigel: The Barite Industry in Missouri. *Trans.* A.I.M.E. (1929) **85**, 256.

W. A. Tarr: The Barite Deposits of Missouri. *Univ. Missouri Studies* (1918) **3**, 101–102.

R. M. Santmyers: Barite and Barium Products. U.S. Bur. Mines *Inf. Circ.* 6221 (1930) 16–18. (Flow sheet needs revision.)

G. J. Young: Milling Barite in California. *Eng. & Min. Jnl.* (1930) **130**, 70.

For most of its uses barite of high purity is demanded. It follows that deposits that fail to yield a fairly white, high-grade product by the foregoing methods could not be worked. Within limits, adhering quartz and limonite could be removed by cobbing, and acid bleaching of off-color ore has been practiced to some extent, but ores containing any considerable amount of mixed grains have remained unsalable. Even in Missouri, which produces roughly half of the domestic output, some difficulty is experienced in disposing of the product in competition with foreign barite because the typical grade is 93 per cent $BaSO_4$ and up to 1 per cent iron, whereas a considerable premium might be offered for a product containing 97 to 98 per cent $BaSO_4$ and a very small amount of iron. Flotation appears to afford a solution of this problem and may be applicable not only as a primary process for beneficiating ores containing intergrown silica or fluorspar but also as a cleaning process in central plants receiving jig concentrates and lump from various sources. The Bureau of Mines is working on the recovery of salable barite from mixed ores of the Carolinas and other southern states.

A substantial part of the consumption of crude barite is pulverized and employed in the form of ground barite for rubber, paper, and other uses, chiefly as a filler. Raymond roller mills may be used for this purpose.

BAUXITE

Bauxites can be freed from silica by running them through a log washer if the silica is in the form of claylike minerals, but many bauxites cannot be concentrated by ordinary washing. Sandy overburden ordinarily is carefully stripped and finally swept off before mining begins. The clean ore then needs only to be crushed to a suitable size (under 1½ or 2 in.) for calcining in rotary kilns (around 1100° F.) to save freight and facilitate fine grinding at the chemical purification plant; a small amount is dead-burned (around 2000° F.) for use in abrasives and refractories. In British Guiana[6] the ore crushed under 3-in. size is cleaned in Dorr washing units, oversize of trommel (⅜ in.) and rake product being saved and dried in rotary kilns requiring 80 lb. of coal per ton.

BENTONITE

Bentonites generally are marketed without previous preparation, lump material being shipped to plants in consuming centers, where it is ground or blended as desired. Dilution with waste or overburden is avoided in mining. Most bentonites do not become gummy until the moisture content exceeds 30 per cent, but generally they are dried for several days before shipment or milling. Preliminary reduction to about ⅜ in. may be in rolls (arranged to operate at different speeds to develop tearing action) or in special cutting boxes, after which the material goes to mechanical driers heated to around 250° F. (gel-producing properties may be injured at over 400° F.). The dried material being quite friable, it is preferably ground by attrition instead of impact; ball mills, slug mills, rod mills and roller mills are all employed. Despite simplicity of operation, total cost of mining and milling has been reported as $15 a ton, of which $3 is for crushing and drying alone.

CEMENT

Portland cement is produced by incipient fusion of a chemically controlled mixture of pulverized materials—typically four parts limestone and one part clay—to form a clinker, which is crushed (½ in.), blended with raw (rarely calcined) gypsum as retarder, and finely ground (75 per cent or more through 325 mesh). At the raw end, wet grinding is occasionally employed, but dry grinding is practiced at 80 per cent of the American plants. Hardinge mills, tube mills, Raymond mills, and various other mills are employed at both raw and clinker ends. A new type of grinding mill, consisting of superimposed sets of ball bearings riding in separate rings, has recently been developed by the Babcock & Wilcox Co. Closed-circuit grinding, only recently borrowed from ore-dressing practice, has greatly increased grinding efficiency at cement plants, both wet and dry. Modern practice tends toward progressively finer grinding. An enormous amount of power is expended in grinding at cement mills, but as waste heat power is generally available power economy is of secondary importance to fuel economy, which now receives more attention than possible improvements in grinding technique. U.S. Patent 1931921 covers flotation of calcite for use in cement manufacture. By dropping quartz and other coarse particles by this process, the Valley Forge Cement Co.[7] is now able to make a better burned and more uniform clinker with less fuel, and also to utilize cement rock reserves that previously were considered unavailable.

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[6] L. Litchfield, Jr.: Bauxite Mining in British Guiana. *Eng. & Min. Jnl.* (1929) **128**, 347–9.

CHALK[8]

Chalk, pulverized, purified and carefully sized, is the ordinary whiting of commerce. Whiting substitutes consist of finely ground limestone or dolomite, marble flour, white marl, clay, and chemically prepared $CaCO_3$ (precipitated chalk). Domestic whiting requirements are supplied by about twelve grinding plants employing European raw material. In the older types of mills the chalk is ground wet in edge runners after flint nodules have been picked out, and the slurry goes to a series of settling tanks, the first removing sand, which is discarded, the second producing the lowest grade ("commercial"), and subsequent tanks producing intermediate grades ("gilder's" and "extra gilder's") and the finest product (Paris white). The overflow from the last tank is returned to the mill circuit. More modern mills use bowl classifiers, hydroseparators and thickeners, followed by filters and driers; this method yields but one grade of whiting at a time but is more economical and furnishes a very uniform and dependable product. In Kent, England, the chalk is broken up and kept in suspension in a circular stone pit, 15 ft. in diameter, provided with a central vertical shaft, fitted with four arms, from which short sections of rail are suspended by chains; the slurry overflows through a ⅛-in. screen and then is washed through 180-mesh trommels (sand and coarse chalk sold for asphalt surfacing) before being led into settling pits. There are four sets of these, with eight pits to each set. Each pit is 10 by 6 by 8 ft. deep. The slurry enters the first pit, overflows to the second, and so on, until all eight are full, after which the stream is diverted to another set of pits. The settled whiting is dug out by hand and carried to drying floors paved with heated iron plates, where a 10-in. sludge takes 36 hr. to dry. The dried material is fed to a swing-hammer disintegrator and bolted; the oversize of the bolting trommel returns to the disintegrator.

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[7] N. C. Rockwood: Chemistry Applied to Cement Manufacture. *Rock Products* (1934) **37**, 32–37.

[8] H. S. Spence: The Manufacture of Whiting in England. *Canadian Min. Jnl.* (1926) **47**, 907.

A. B. Parsons: Dressing Crude Chalk by Modern Methods. *Eng. & Min. Jnl.* (1923) **116**, 415–420.

O. Bowles: Chalk and Whiting. U.S. Bur. Mines *Inf. Circ.* 6482 (1931) 8–9.

CLAYS[9]

Clays used for the manufacture of common brick and other heavy clay products receive little or no preparation before use, other than the thorough mixing and perhaps crushing that they receive in the manufacturing process. Fireclays, low-grade stoneware clays, and other clays valued at under about $3 a ton likewise undergo no beneficiating treatment, the quality being maintained ordinarily by selective mining. Only high-grade clays—comprising principally china clay but also ball clays, slip clays, and some miscellaneous varieties—are sufficiently valuable to bear the cost of purification and separation into the various technically controlled grades that the market is beginning to specify. Certain high-grade sedimentary kaolins, notably those in South Carolina, contain few or no impurities and formerly were shipped in lump form, in bags, or in bulk, but now consumers in the paper, rubber, and sundry other consuming industries demand a ground product and therefore even these clays are frequently milled by the miners.

Wet washing is the natural method of eliminating sand, small stones, and other foreign matter from residual clays that are mined hydraulically and it is still employed for sedimentary or transported kaolins that are mined by other methods. However, dry processes embodying air separation generally afford a cheaper and often superior means for beneficiating as well as sizing and grading clay. Raymond roller mills or screen mills and various air classifiers are increasingly employed in the clay industry, as well as similar equipment from other manufacturers. Wet methods are exemplified by established practice in Cornwall; the clay-sand stream from hydraulicking is run into sand pits before being pumped to the surface, where it passes through a series of riffled troughs (drags) followed by a series of mica troughs, each 1 or 2 ft. wide and, say, 9 in. deep, arranged in batteries so that the slurry flows slowly (grade about 1 in. in 50 ft.)

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[9] F. R. Van Horn: China Clay. *Eng. & Min. Jnl.* (1930) **130**, 525.

A. L. Curtis: Mining English China Clay, pt. 1. *Sands, Clays, and Minerals* (1933) **1**, 13–19; also *Canadian Chem. & Met.* (June, 1933) 121–122.

C. E. Curtis: The Electrical Dewatering of Clay Suspensions. *Jnl.* Amer. Ceramic Soc. (1931) **14**, no. 3, 219–263.

A. S. Watts: Mining and Treatment of Feldspar and Kaolin in the Southern Appalachian Region. U.S. Bur. Mines *Bull.* 53 (1913).

R. T. Stull and G. A. Bole: Beneficiation and Utilization of Georgia Kaolins. U.S. Bur. Mines *Bull.* 252 (1926).

I. E. Sproat: Refining and Utilization of Georgia Kaolins. U.S. Bur. Mines *Bull.* 128 (1916).

for a distance of 250 ft. or more. In North Carolina, the slurry is raised to the surface in bucket elevators, but otherwise the process is similar except that the settlings in the mica troughs are subsequently utilized for the production of "ground mica." Clay mined otherwise than hydraulically may be converted in a blunger, beater, log washer, or other suitable disintegrator into a slip or water suspension (say 9:1), which may then be freed from grit and mica in much the same manner. Modifications include the introduction of the whirlpool classifier (designed by Stull and Bole, of the Bureau of Mines) ahead of the mica troughs, Dorr equipment (especially the bowl classifier), and screens. At older plants the slurry is all passed through bolting cloth (nominally 160 mesh but pitched 80°). Sand wheels and drag or rake classifiers are employed occasionally for disposing of coarse sand, but ordinarily the sand is simply shoveled out of the various settling boxes and troughs at intervals of a few hours. After removal of impurities, the slurry may be treated with bluing and then pumped to a settling tank or thickener before filter pressing. The filter cake, containing 25 per cent moisture or more, may be air-dried on drying floors or on steam-pipe racks; rotary driers, spray driers, and car tunnel driers are found at different plants. Sometimes the filter cake is passed through a pug mill or auger machine to form a hollow cylinder or other shape that can be dried quickly.

An electrolyte, generally sodium carbonate, may be employed to deflocculate the clay and hasten settling of sand, but it must be neutralized with acid before thickening and filter pressing. Coagulating with alum is possible only when water is not reclaimed; even a small amount of residual alum greatly reduces the benefit derived from the use of deflocculants.

Electrical means (electro-osmosis or cataphoresis) have been employed in Europe for dewatering clays but cooperative work of the Bureau of Mines and the University of Washington shows little gain in efficiency as compared with filter pressing except on very fine-grained clays. The process may be made continuous but installation costs and power consumption are relatively large.

Cryolite

Cryolite occurs in commercial quantity and is mined only at Ivigtut, Greenland. Purification plants for hand-sorted high-grade material are located in Denmark and the United States, and the method employed in this country is of interest as an unusual type of flotation cleaning known as "boiling-over" concentration. Crude cryolite is crushed and treated by Wetherill magnetic separators to remove siderite, then by Wilfley tables to remove sulfides—galena, sphalerite, chalcopyrite and pyrite—but slimes from this process were formerly wasted. Several thousand tons of dark and valueless accumulated slimes, too fine for tabling, eventually were converted into a pure-white product worth up to $260 a ton, simply by boiling in the presence of acid and without the use of additional reagents. Coal, coke, pyrite, sphalerite, chalcopyrite, galena, and miscellaneous debris rise to the top and are skimmed; the siderite and perhaps part of the galena may dissolve more or less prior to removal, depending upon concentration of acid maintained. The process has been further described by Darlington,[10] as follows:

> The screened fine cryolite, passing 80-mesh, with about one part of water to two or three parts of cryolite, is delivered to a boiling vat heated by a coil of extra-heavy lead piping. The vats are of cypress, 6 ft. in diameter by 7 ft. high. The unlined wood lasts several years; it is then lined with lead. Simple boiling brings to the surface a large quantity of organic dirt, which is skimmed. Then several buckets of acid are added, and the tanks boil over. The foam, 2 to 3 ft. thick, is skimmed by means of 20-in. scoops of monel-metal fly-screen; it is then accumulated in settling vats, smelted for lead, copper and silver, or discarded. Boiling is repeated in a lower tank, to insure a good extraction; the product is sent to a vacuum filter; after washing, it is dried in shelf driers.

A few drops of linseed-oil paint added to the contents of a large tank filled with foam will kill the foam, but the antifoam effect of paint or new wood (as in starting a new vat) may be counteracted by a "dope" made by boiling 8 parts of old oil, 1 part of Fels-naphtha soap, and 1 part of water. The old oil is presumably neat's-foot oil from machines employed for cutting and threading old acid pipes; new oil is less satisfactory. An ounce of this "dope" produces a wonderful foam and in starting a new plant it is impossible without it to get foam for several days.

DIATOMITE[11]

Diatomite deposits may be divided into three types: (1) ancient marine beds, compact and more or less massive but sometimes accompanied by seams and partings of clay; (2) fresh-water lake and stream deposits, generally of more recent origin; and (3) deposits in lakes and swamps where the process of accumulation is even now going on.

The immense deposits of marine diatomite found along the coast of Southern California afford the bulk of the domestic tonnage. At

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[10] H. T. Darlington: Boiling-over Concentration. *Min. & Sci. Pr.* (1922) **124**, 217–218.

[11] R. B. Ladoo: Non-Metallic Minerals. Occurrence—Preparation—Utilization, 190–198. New York, 1925. McGraw-Hill Book Co.

P. Hatmaker: Diatomite. U.S. Bur. Mines *Inf. Circ.* 6391 (1931).

Mining and Milling Diatomaceous Earth in California. *Eng. & Min. Jnl.-Pr.* (1925) **119**, 970.

R. Calvert: Diatomaceous Earth. New York, 1930. Chemical Catalog Co.

V. L. Eardley-Wilmot: Diatomite, Its Occurrence, Preparation, and Uses. Canada Dept. Mines, Mines Branch, Ottawa, *Pub.* 691 (1928) pt. 5, 18.

Lompoc, Calif., special attention is given to securing a large proportion of large, sound blocks that can be split and sawed into suitable sizes for brick and other forms. Where a pulverized product is required, common practice in California, Nevada, Oregon, Washington, and elsewhere is to dry the product, by natural means if local conditions permit, and pass it through crushers or toothed rolls in closed circuit with a trommel, the undersize of which passes to some form of pulverizer, generally of the swinging hammer type. The object of pulverizing is to free the individual diatoms, and care must be taken to avoid destroying the structure. The value of the product for most purposes depends upon the dead-air spaces formed by the hollow cells which account for the low apparent density of the material (frequently less than 16 lb. per cubic foot for dry, loose powder) and for its heat-insulating and sound-insulating properties. The material from the pulverizer is classified into the various fine sizes in a series of cyclones, which may be preceded by sand traps for removing objectionable impurities. The material from the cyclones is sacked for market, the sacks sometimes being passed between light rolls to reduce their bulk, so that more of them may be loaded into a freight car.

A novelty at the Trio-O-Lite Co. mill, Carlin, Nev., is the use of a single-stamp mill with a series of prongs to force the material through the 1-in. spaces of the grizzly.

Calcination may be necessary for some diatomites and for some uses, even though the amount of carbonaceous matter is small. To remove the combined water, a full red heat of about 800° C. is the most practical temperature. In all diatomite high in iron, either with or without the addition of a reducer, the lower the calcining temperature the whiter will be the product. In some cases, perhaps, iron stain may be avoided by adding about 5 per cent of salt before calcining. Soda generally yields little improvement, and while lime may prevent red color the quantity required may mar the purity of the product.

The lake or swamp type of deposit usually contains more or less organic matter that has not completely decomposed; certain Florida deposits, for example, are virtually in peat bogs, the material being taken from shallow lake bottoms by dredges. The dredge product is unwatered, air-dried, and finally run through furnaces to burn out the organic matter, which often constitutes 50 per cent or more of the drier product. Subsequent pulverizing involves no special features. In the Adirondacks, a deposit is mined by a clamshell bucket mounted on a floating hull and loaded into a car that stands on another small scow. Water is squeezed out in a small press, and the cake is dried in outdoor racks for two or three weeks before calcining in an open kiln. Attempts to remove clay have been made at various properties but generally are incomplete and costly.

FELDSPAR[12]

The concentration of feldspar has been almost entirely a matter of hand picking. This appears to be the only means of separating potash and soda spar, and so far at least it is the only feasible commercial means of separating feldspar from quartz, which has practically the same specific gravity. Bureau of Mines experiments indicate the possibility of separating quartz and feldspar by flotation, but complete reports on tests are not available, and the economics of the problem calls for serious attention in view of the low price at which clean lump spar of good quality can be obtained. The field for magnetic purification is already well established, reclaiming for ceramic use thousands of tons of otherwise valueless material, developing new uses in the glass industry, and providing pottery and glass makers with a far better product. Feldspar is marketed chiefly in ground form, and before the introduction of improved methods of magnetic treatment it was impossible to employ jaw crushers, steel rolls, or other equipment that might introduce iron into the product by abrasion. Manganese-steel wearing parts have also been a factor in eliminating iron contamination. At almost all feldspar operations, however, there is still an appalling waste in the form of material below hand-picking size. The discharge from picking belts, amounting often to 50 per cent of the mine output, either goes to waste or is used for road surfacing and otherwise as crushed stone. Further wastage occurs as a result of breakage in handling, for the spar is loaded with forks, and fines are discarded practically up to the time that the material is charged to the final pulverizing mill. At smaller operations, the only beneficiation is sorting and cobbing at the mine, but modern tendency seems to favor sending quarry-run material over a grizzly (2 to 5-in. spaces), crushing oversize, scrubbing or otherwise washing material over ½-in. screen, and passing a fairly well sized product over picking belts. At the well designed plant of the Tennessee Minerals Products Co., Spruce Pine, N.C., a crew of 16 women or girls and 3 men remove 2½ to 4 tons of material from 7 to 8 tons run hourly over the belt. One man is foreman, the other two are cobbers. Six products are picked in the following order: White flint, No. 1 potash spar, No. 1 soda spar, No. 2 potash spar, No. 2 soda spar, and mica. Large pieces of No. 1 spar are picked out at the apron feeder in advance of the grizzly above the crusher. Large blocks of mica are the only product sorted at the mine.

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[12] B. C. Burgess: Methods and Costs of Milling Feldspar at the Minpro Plant, Tennessee Mineral Products Corporation, Spruce Pine, N.C. U.S. Bur. Mines *Inf. Circ.* 6488 (1931).

O. Bowles and C. V. Lee: Feldspar. U.S. Bur. Mines *Inf. Circ.* 6381 (1930).

H. S. Spence: Feldspar. Canada Dept. Mines, Mines Branch *Bull.* 731 (1932) 105–130.

While magnetic purification has more general applications in removing iron-bearing impurities, probably its most noteworthy use in the feldspar industry is in the production of the coarser granular grade desired by the glass trade. For the ordinary grinding of feldspar, pebble mills are universally employed, but the "glasspar" product of the Tennessee Mineral Products Co. is reduced by graded crushing in rolls. From the surge bin the material, previously reduced to about ½ in. in a Reliance crusher, is chuted to Sturtevant rolls set about ⅜ in. apart. The discharge from these rolls is elevated to a double-deck Hum-mer screen with ¼-in. and 20-mesh screens. Oversize on the coarser screen goes back to the rolls; the intermediate size goes to a second set of Sturtevant rolls set $\frac{3}{16}$ in. apart; and the undersize through 20 mesh, which is now finished as to maximum size, is conveyed to a Gayco air separator for removal of fines. The discharge from the second set of rolls is elevated to a second double-deck Hum-mer screen with 8 and 20-mesh screens, which yields an oversize returned to the second set of rolls, undersize sent to the Gayco separator, and an intermediate product which goes to a third set of Sturtevant rolls set close. A third Hum-mer screen with 10 and 20-mesh screens takes the discharge from the last set of rolls, the oversize that consists principally of mica being a byproduct and the intermediate size being returned to the rolls. The 20-mesh undersize from all three sets of Hum-mer screens is put through the Gayco separator to eliminate minus 200-mesh material. The Gayco oversize is split into two sizes at 40-mesh to be run separately through two Johnson induction separators. Even muscovite without visible iron stain is removed in these machines, and the product consistently contains less than 0.06 per cent iron, or about one-half the usual maximum specification for this type of spar. Comparative screen analyses show a preponderance of sizes coarser than 48 mesh for this method of reduction, compared with scarcely 11 per cent in this range and over 50 per cent minus 200 mesh in the product of the Hardinge mill unit.

The fine grinding of feldspar for ceramic purposes (200 mesh is a common specification) has likewise been revolutionized during the last decade. Formerly the almost universal practice was to use stone chaser mills for fine crushing, followed by grinding in batch pebble mills. The stone chasers, which are still employed at a few older mills, consist of two large-edge runner stones of dressed quartzite, or granite, about 6 ft. in diameter and 20 in. wide, which are revolved about a vertical drive shaft and over a bed of stone blocks. The crude lump spar, broken by hand sledges, is shoveled under the stones and the product is returned until it will pass 12 or 14 mesh. Old-style batch mills, with charges of 1½ or 2 tons and run for 5 to 8 hr. according to the degree of fineness desired, are likewise still in use, but probably 95 per cent of the domestic production is now ground in continuous (chiefly Hardinge) mills, preceded by jaw crushers or jaw crushers and coarse rolls, which reduce the material to about ½ in. All grinding mills, whether of batch or continuous type, are lined with flint or Silex and employ French or Danish flint pebbles for grinding. Air-swept mills are used at some plants, but other operators do not favor them because the market desires a considerable production of ultra-fine sizes. Closed-circuit grinding with an air separator is the rule, but the oversize from the air separator may be reground in a tube mill. Drying is practiced at few plants, but even when the moisture content of the air-dried lumps introduces no appreciable mechanical difficulties, increased efficiency may result from drying, and at one plant at least the removal of dust (consisting mainly of mica, clay and other impurities) in the drying operation effects an improvement in product sufficient to offset the cost of drying.

Wet grinding is practiced in England but not in this country; although costly, it is supposed by some to provide a better quality of "grain."

Fine grinding of feldspar costs at least $1 a ton at most American plants. Total milling costs covering 34,108 tons treated by a North Carolina mill in 1929 amounted to $1.45, of which $0.83 was allocated to fine grinding and finishing. Grinding costs varied according to limiting size of product from $0.633 for 20-mesh material to $1.101 for 200-mesh and $1.370 for 230-mesh.

Cost of grinding, however, is a relatively small factor in determining the selling price of feldspar, being overshadowed by general expense and scarcely more important than selling expense or capital charges. It follows that measures taken to reduce sales resistance and increase the ratio of output to capacity are of prime importance. By careful laboratory control combined with stocking of numerous grades to permit blending to desired specifications, operators are better able to cater to customers' specialized demands, and gains in volume of business thereby have offset the increased cost of plant and direct operating expenses.

FLUORSPAR[13]

Residual deposits may yield metallurgical grades of gravel spar simply by crushing and running the material through a log washer, the fluorspar occurring in small pieces in a matrix of clay and coil (weathered limestone), but such deposits are mostly worked out. Except at some of the smaller mines, where only hand sorting is done, the bulk of the fluorspar produced in the United States must be subjected to a fairly complex concentrating process, which in effect starts at the mine. As much waste as possible is left underground, additional waste is sorted out at the shaft "ore" pockets, and even in the stopes blasting is done with light shots to avoid excessive fines. At the mill, material over ¾ in., after being cleansed with water jets, is fed mechanically to a picking belt from which acid spar (generally 98 per cent plus CaF_2), No. 1 and No. 2 lump, and occasionally optical crystals are removed by hand and placed in suitable bins. Coarse waste, amounting in the Illinois-Kentucky field (1925) to about 16 per cent of the total mill feed, is picked off at this point, as well as tramp iron if no magnetic pulley is provided. About 5 per cent (1925) of the mill feed is recovered as sorted lump material, some of which is sold as such, and the remainder is dried on steam coils, ground and screened for the enameling or glass trade. The further mechanical treatment is mainly for the recovery of fluxing grades of gravel spar, although flotation concentrates are generally of acid grade, and some high-grade concentrates suitable for grinding may be drawn from the jigs.

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[13] E. C. Reeder: Milling Methods and Costs at the Hillside Fluorspar Mines. U.S. Bur. Mines *Inf. Circ.* 6621 (1932).

The discharge from the picking belt is crushed by gyratory or jaw crusher to under 1 in. and reduced below ¾ in. by rolls before joining the undersize that bypasses the picking belt. The pulp may be dewatered at this point by a chain drag, the overflow going to a sludge pond. After being screened, all material minus ¾ in. and over about 2 mm. is carefully separated into perhaps four or five different sizes, each size going to a separate jig. The jigs are mostly of the Harz type with five cells and special draw-offs because of the amount of screen concentrate. Large jig beds and fine screens are used to reduce the amount of hutch product. Sand sizes under 2 mm. are classified hydraulically and treated on tables. High-grade jig concentrates can be made only from high-grade feed. Tailings and low-grade ore[14] can now be handled by preliminary treatment in a rougher jig. Hancock jigs have been employed for this purpose, followed by Harz jigs and, for minus 8-mesh material, Deister Plat-O tables, as in former practice.

Galena accompanies much of the ore in the Illinois-Kentucky field and is partly recovered on the first cell of the jigs, but as it is brittle more of it comes on the tables. At one mill the first set of four tables produces lead concentrates, fluorspar concentrates, and a middling which is reclassified and passed to a second set of tables where lead, fluorspar and waste tailing are produced.

The foregoing description relates to gravity separation chiefly in the Illinois-Kentucky field, but milling practice throughout the West embodies the same principles. As compared with jig and table concentration of metallic ores, fluorspar milling differs in three principal features: (1) of primary importance, is the high purity required of the product; even gravel spar should contain only 5 to 15 per cent gangue. Barite, galena, sphalerite, pyrite, all other sulfides or sulfates, all other lead or zinc compounds, and (for ceramic grades) iron compounds, are highly injurious impurities and must be practically eliminated from the product. (2) The proportion of concentrates represents a very large proportion of the total mill feed (average, 60 per cent in 1925); in calcite gangue, ore containing 50 per cent spar has generally been considered about as low as can be worked, and for certain classes of siliceous ores a minimum of 75 per cent fluorite is ordinarily considered necessary in order to obtain a clean product, unless the proportion of included grains is extraordinarily low. (3) There is very little difference in density between fluorspar and its accompanying minerals; fluorite has a specific gravity of 3 to 3.25, which allows only a meager margin for separating it from calcite (sp. gr. 2.7) or even quartz (sp. gr. 2.6 plus).

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[14] E. C. Reeder: Depression Developments in Fluorspar Milling. *Eng. & Min. Jnl.* (1934) **135**, 301–4.

Owing to the difficulty of separating fluorspar from calcite or quartz by gravity means, many other methods of concentration have been tried. Electrostatic cleaning has been recommended as feasible, but the most unusual innovation is a decrepitation process which was used fairly successfully for several years in British Columbia, and to some extent in New Mexico, where the gangue is siliceous and where no considerable amount of other decrepitating mineral, such as barite, is present. In some ores fluorspar breaks into finer particles than the associated minerals during the ordinary processes of mining and crushing; consequently selective screening alone may result in a considerable concentration. When heated to around 1200°F. fluorspar flies to pieces, freeing itself from adhering gangue, most of which can then be screened out. By using a fine screen, 40 mesh, the Fluorspar Mines of America, in New Mexico, obtained a good product but at the expense of heavy losses. In 1931, initial shipments of ceramic spar were made from the plant of the Alamo Fluorspar Mills, near Derry, Sierra County, New Mexico, which employs decrepitation and screening to beneficiate the product before grinding.

The Rock Candy mill of the Consolidated Mining & Smelting Co., at Trail, B. C., was described by Freeland in the Annual Report of the Minister of Mines of British Columbia for 1919 (Vancouver, 1920, 164–165). The ore was reduced by stage crushing, dried, and graded by impact screens into three sizes, which were fed separately to rotary kilns 14 ft. long and 3 ft. 2 in. in diameter, making 3 r.p.m. Gases from these kilns pass through the drier. The feed for No. 1 kiln was plus ¼ inch, No. 2 kiln being minus ¼ inch plus 8-mesh, and No. 3 kiln being minus 8-mesh plus 15-mesh. Best results were obtained on the smallest sizes treated in No. 3 kiln, and the undersize of the 15-mesh screen was not decrepitated at all; this middling product contained only about 5 per cent silica and was used locally for making acid or for adjusting shipping concentrates to specifications. It was never possible to remove sulfides, and the grade of the product was rarely wholly satisfactory. Moreover, recoveries remained low even after Wilfley and Deister tables were added to the mill to treat the fines. A maximum of 7477 tons of concentrates was produced in this mill in 1921, and subsequent operations were impeded by the tariff on shipments to the United States which became effective the following year.

The flotation of fluorspar ores is the subject of U. S. Patent 1785992, issued Dec. 23, 1930 to J. C. Williams and O. W. Greeman, assigned to the Aluminum Co. of America, of which the Franklin Fluorspar Co. is a subsidiary. Preliminary studies by the Bureau of Mines on fluorspar flotation have been reported by Coghill and Greeman.[15] The Franklin mill at Rosiclare, Ill., came into commercial production of acid-grade concentrates by flotation on March 18, 1929. According to Hatmaker[16] the pulp at this mill is reduced to minus 65 mesh and slimes (minus 325 mesh) are removed, colloidal material having an adverse influence on recoveries. Four reagents are employed—a depressant, a collector, a frother, and a froth conditioner. Pulp consistency averages 1 part ore to 7 parts water. The collecting and frothing agents are added in the conditioner and depressing and froth-conditioning agents in the flotation machines. Lead, silica, and calcite are depressed, and fluorspar is floated. The pulp is kept warm. Recoveries vary according to the character of the feed.

Flotation has been used at several plants to remove sulfides, generally after preliminary gravity treatment to eliminate lighter gangue minerals.

FULLER'S EARTH[17]

Proper grain size is of prime importance in preparing earths for decolorizing and filtering purposes. Coarse sizes (especially 15 to 30 mesh, 30 to 60, and 40 to 60) are demanded by petroleum refiners, and the various grades of finer sizes, ranging from 60 to 300 mesh, are employed by manufacturers of edible oil. The raw clay, roughly dried if necessary, is crushed in two stages to under 1 in., generally in corrugated or toothed rolls, before entering oil-fired rotary driers. This "roasting" is carefully controlled according to the nature of the earth and product desired; ordinarily it takes 12 to 20 min. to pass through the drier, and typical temperatures are 600° to 700° F. At some plants, the hot earth is stored in cooling tanks; at others it goes direct to the secondary roll system, where it is further reduced in size and screened. Since attrition tends to produce undesirable fines, rolls, roller mills, hammer mills, and impact pulverizers are usually preferable to buhr mills, tube mills, etc. Common practice is graded crushing in corrugated rolls followed by screening on vibrating screens and bolting through Swiss silk in gyratory sifters. A Florida concern uses a Raymond automatic pulverizer with throw-out attachment for eliminating sand from poor-grade earth that formerly was thrown away.

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[15] Coghill, W. H. and Greeman, O. W.: Flotation of Fluorspar Ores for Acid Spar: U.S. Bur. Mines *Rept. of Investigations* 2877 (1928).

[16] P. Hatmaker: U.S. Bur. Mines. Private communication.

[17] P. Hatmaker: Fuller's Earth Chapter, Mineral Resources of the U.S., 1931, pt. 2, 87–88. U.S. Bur. Mines.

P. G. Nutting: The Bleaching Clays. U.S. Geol. Survey *Circ.* 3 (1933).

GEMSTONES[18]

Precious and semiprecious stones are derived principally from alluvial deposits and are recovered by placer-mining methods. Labor being cheap at most producing localities, hand washing in miners' pans or bateas is common, and frequently the washed gravel is simply picked over carefully on the ground or on a table. Jigs are employed occasionally for coarse gravel. The specific gravity of rubies and sapphires (corundum) is around 4, and the diamond and sundry other gems are considerably heavier than quartz, but mechanical methods of separation are suitable only for preliminary concentration to eliminate the huge quantities of waste without attempting to produce a clean concentrate. Mechanical concentration is employed on a large scale only in the Kimberley district of South Africa. Near the surface the diamond-bearing rock was soft, and diamonds were recovered readily by washing. As the workings become deeper and "blue ground" was encountered, it was found necessary to allow the freshly mined rock to weather for 4 or 5 months before sending it to shallow iron pans for preliminary concentration. To avoid the tie-up of capital while rock was lying on the floors disintegrating by natural processes, stage crushing of the fresh rock was developed so as to prevent excessive destruction of diamonds. Diamonds are easily broken by a sudden blow, but there is little loss in rolls equipped with springs or in pebble mills. The rotary pan is an efficient machine of large capacity (9 cu. yd. per hour) for low-grade concentration; its overflow removes all but about 1 or 2 per cent of the mill feed, and the concentrates are further reduced to a small fraction of 1 per cent of the original volume in pulsator jigs. These are mostly of a special type (de Beers) using a ragging or bed of material heavier than the diamond;

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[18] P. A. Wagner: Diamond Fields of Southern Africa, 200–240. Johannesburg, S. Africa, 1914. *Transvaal Leader.*

A. F. Williams and J. Harbottle: Present Day Practice of Diamond Mining, Including Recovery. *Proc.* Third (Triennial) Empire Min. and Met. Congress, Johannesburg, S. Africa (1930) **3**, 1–20.

J. Sim: The Rotary Pan for Diamond Concentration. *Min. Mag.* (1933) **48**, 215–216.

D. Draper: The Evolution of Diamond Mining and Winning Machinery in South Africa. S. African *Min. & Eng. Jnl.* (1928) **38**, 521.

concentrates are made only through the screen and are removed from the hutch by a bucket elevator. Light minerals having now been largely eliminated, the diamonds (and zircons) are caught on grease tables, which are hung on flexible supports and are oscillated sideways like a Frue vanner. The table deck may be an endless rubber belt provided with devices for applying the grease (petroleum jelly) and later scraping it off with the adhering diamonds, or simply a galvanized iron tray, which can be removed and cleaned periodically. The grease-table product is put into perforated containers and lowered into boiling water. The melted grease rises and may be skimmed off for reuse; diamonds smaller than the perforations are periodically removed from the vat where the boiling is done. The diamonds are next washed with hot caustic solution and finally treated with hydrofluoric acid. The stones are roughly graded and made up into parcels at the mines.

GRAPHITE[19]

Flake graphite is invariably associated with gangue that must be removed by a suitable cleaning process. Amorphous graphite deposits are generally fairly pure; in Ceylon vein graphite is cobbed with hatchets and wiped carefully by hand, and smaller sizes are cleaned by primitive methods of panning and winnowing that could not be applied commercially except with abundant and cheap coolie labor. Prior to the introduction of flotation the concentration of flake graphite afforded almost insurmountable difficulties, and even yet the production from domestic ores of a concentrate that consumers will buy is a problem that has not been solved commercially. There is little demand for a product containing less than 85 or 90 per cent of graphite carbon, and to complicate matters the structure of the flake appears to be more important than chemical purity. Even screen analysis fails to reveal the requirements of crucible makers who demand a tough flake as well as a fairly large one free from impurities naturally embedded between the laminae or pressed in during the milling process (pancaking). The fact that the specific gravity of graphite (2.2) is only a trifle less than that of associated impurities does not favor gravity methods of separation, and purely mechanical dependence upon the flaky character of the mineral in tabling, screening, or classification in water or air has not resulted in satisfactory recoveries or grade of product. Electrostatic separation, though possessing many of the disadvantages of purely mechanical processes, has been moderately successful, especially as a final cleaning or refining process.

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[19] B. W. Gandrud, G. D. Coe, C. S. Benefield and I. N. Skelton: The Flotation of Alabama Graphite Ores. U.S. Bur. Mines *Rept. of Investigations* 3225 (1934).

R. Sauffrigon: Le Graphite; Etude Technique (Sommaire) *Mines Carrières Grandes Entreprises* 132 (October, 1933) 5–14.

Black Donald Graphite Operating New Hydro-Electric Plant and Concentrator. *Eng. & Min. Jnl.* (1929) **128**, 368.

W. Thompson: Methods of Treating Madagascar Graphite. *Eng. & Min. Jnl.* (1930) **130**, 4–6.

H. S. Spence: Graphite. Canada Dept. Mines, Mines Branch, *Rept.* 511 (1920).

G. D. Dub and F. G. Moses: Mining and Preparing Domestic Graphite for Crucible Use. U.S. Bur. Mines *Bull.* 112 (1920).

C. S. Parsons: The Concentration of Flake Graphite Ores. Canada Dept. of Mines, Mines Branch Mem. Ser. (1926) No. 25.

P. M. Tyler: Graphite, General Information, I. U.S. Bur. Mines *Inf. Circ.* 6118 (1929).

H. B. Menardi: Modern Flotation Plant for Graphite. *Rock Products* (1928) **31**, 74–77.

W. G. Hubler: Concentrating Graphite in Southern California. *Eng. & Min. Jnl.* (1928) **125**, 1059–60.

R. von Schoen: Aus der Praxis der Flotation von Graphite. *Metall u. Erz* (1932) **29**, 181–182.

The earthy residual ores of Madagascar are shoveled into a sluice where the graphite apparently behaves very much like mica in the mica troughs used in clay washing. In this preliminary process (*debourbage*) the sand is largely settled out, and in the last troughs the slurry is passed over a series of metal screens which remove various sizes of material. Losses in this operation are said to be very high, particularly in large flake, and the product contains only 45 to 60 per cent carbon and needs to be cleaned. The natives raise the grade to 80 per cent or more by winnowing and screening the dried product. Flotation is employed at one or more plants, and further treatment (electrostatic, chemical, etc.) generally is necessary to eliminate mica and other troublesome impurities. At the Black Donald mine, Ontario, Callow cells combined with Wilfley and Deister tables are employed to produce small flake, "amorphous," and dust graphite of salable grade from a mine product containing 65 per cent or more of graphite. K. & K. machines have been used in at least three domestic mills with apparent technical success, the cleaner concentrate from flotation being further cleaned on tables, electrostatic machines, buhr mills, and screens, or by alkali fusion. Callow cells and Minerals Separation machines have been used in Alabama. The main problem is to free the graphite from gangue without destroying the flake and to remove sand as quickly as possible. Rod mills are often less destructive than ball mills, but in the Black Donald mill stamps are employed for secondary crushing. To avoid excessive size reduction, it has recently been proposed to produce a coarse flotation concentrate with about 65 per cent of graphite and remove the silica, some of which is interlaminated, by alkali treatment.

Even after delivery at consumers' plants, most graphite goes through a series of milling operations. Crucible makers, in particular, generally put their graphite through special cleaning processes to obtain the type of material they want for their crucibles and incidentally produce a variety of byproducts which are used in foundry facings, pencil leads, and other manufactures of graphite.

GYPSUM[20]

Gypsum is marketed raw as a land plaster (which is ground to at least 5 mesh, usually to 20 mesh or even to 100 mesh), cement retarder (crushed to ½ in.), terra alba (an extra pure white grade ground to 200 mesh or finer), and a few minor products, but 75 per cent of the domestic output is calcined. Where rotary kilns are employed, the raw gypsum is merely crushed to ¾ in., but the material usually is calcined in kettles. It may be pulverized either before or after calcining, or both. Primary crushing to about 3 in. is in jaw crushers (nippers). Secondary crushing (to ¾ or ¼ in.) may be in rotary crushers (pot crushers or "crackers," which are similar in action to large coffee mills), Edison rolls, or (more recently) hammer mills. Further reduction, generally following removal of tramp iron and drying, is accomplished in buhr mills, horizontal or vertical emery mills, disintegrators or roller mills. Raymond mill installations with air separation are standard equipment in several districts. Gypsum is soft and easily ground even in the raw state, but impurities may be present which favor the use of roller mills and tube mills. Ball mills are of rather recent introduction and are employed chiefly for regrinding calcined material to meet the demand for a better prepared product. In Canada, the limiting screen for raw grind is typically 20 mesh; in New York the grinding is continued to 90 per cent through 100 mesh. Rod mills have been recommended for regrinding, not so much to produce finer sizes as to fluff up the product. Vibrating screens are employed for sizing, but all gypsum equipment tends to "gum up," and air separators are increasingly used. Various admixtures are mechanically blended with calcined gypsum after grinding to control the set and otherwise modify its character.

KYANITE[21]

Kyanite crystals are bladelike and elongated, consequently they tend to be separable by screening methods from rounded or square particles of associated minerals. However, the uses of kyanite in the refractories and fine ceramics industries are such as to demand a high degree of purity, especially as regards freedom from iron, titanium compounds, garnet, and other minerals tending to discolor a fired product or lower its softening temperature. Commercial production has been partly by hand sorting from segregation deposits, but large extensions in use depend upon utilization of crystalline schists and quartzites, which in the Appalachian region may contain from 2 to 20 per cent or more of kyanite over considerable areas.

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[20] The Technology of the Manufacture of Gypsum Products. U.S. Bur. Stds. *Circ.* 281 (1926) 20–42.

F. A. Wilder: Gypsum: Its Occurrence, Technology and Uses. Iowa Geol. Survey (1917–18) **28**, 211–244.

L. H. Cole: The Gypsum Industry of Canada. Canada Dept. Mines, Mines Branch *Pub.* 714 (1930) 98–101.

R. W. Stone: Gypsum Products, Their Preparation and Use. U.S. Bur. Mines *Tech. Paper* 155 (1917).

[21] V. L. Mattson: Progress in the Commercial Development of Kyanite in the South Atlantic States. *Bull.* Amer. Ceramic Soc. (1934) **13**, 226–228.

F. H. Riddle: Minerals of the Sillimanite Group. *Eng. & Min. Jnl.* (1932) **133**, 141.

In Ogilby, Calif., the Vitrefrax Corporation employs decrepitation to reduce the quartz grains to a small sand size, which is readily separated on shaking screens. The ore is first heated in a rotary kiln so as to reduce the iron, which is removed later by a magnetic separator, and the hot calcine is quenched in water, thereby shattering the quartz crystals.

The McLanahan-Watkins Co., near Charlotte Court House, Va., uses simple wet gravity concentration. Following scrubbing in a small cylindrical washer the decomposed overlying kyanite-bearing quartzite mantle yields a mill feed carrying about 50 per cent kyanite crystals. The fresh rock, which carries some pyrite (mostly weathered to limonite), goes direct to a rod mill without preliminary crushing. For initial grinding a light load of rods is employed, which tends to separate the crystals without pulverizing them below about 10 mesh before they go to the tables. Concentrates from preliminary tabling are reground to 40 mesh and finer, using a heavier load of rods, and retabled. The final product contains 90 to 95 per cent kyanite, but further cleaning would be required to eliminate iron and titanium. The ore carries 3 per cent or more of rutile, which is largely removed by the tables.

At Burnsville, N.C., Celo Mines, Inc. uses a dry process involving selective screening, magnetic separation and electrostatic cleaning. After being crushed to ¾ in., the rock is reduced to 20 mesh. Four three-deck Tyler Hum-mer screens in closed circuit with the grinding mill furnish the desired splits for the separators and remove the fines. Fines are low in kyanite and may be rejected; minus 80-mesh material is said to run under 0.5 per cent and minus 60 plus 80-mesh only 7 per cent, whereas coarser sizes may contain 20 per cent or more. The Johnson induction magnetic separator for this job was specially designed by the Exolon Co. Garnet, some biotite, most of the sulfides, and iron from the mill are removed on the first roll of the machine; the second roll takes out most of the remaining biotite; and the third roll completes the biotite separation and removes much muscovite. The magnetic machine concentrates consist principally of quartz and kyanite with a little feldspar, apatite, beryl and the remaining muscovite. The final product from the electrostatic separators, of a modified Huff type, regularly contains up to about 98 per cent kyanite. Some buyers demand a calcined product.

Whereas lump material is usually calcined by stacking in irregular shaped masses in specially designed circular kilns, an oil-fired rotary kiln is employed for the granular concentrate at Celo mines.

Laboratory tests by the Bureau of Mines[22] and others on kyanite-bearing mica schists have indicated the possibility of recovering high-grade mica concentrates as a joint product. Success in table work depends upon close sizing, and the feed should be as coarse as possible. Air separators have been suggested for minus 100-mesh material, in case it contains enough kyanite to justify treatment. Experimental work using dry tables has been encouraging.

Preliminary work with flotation indicates that a definite separation can be made between quartz and kyanite but that serious difficulties develop when other minerals are present.

MAGNESITE

Magnesite is mostly either "dead-burned" (over 1450° C.) or calcined to caustic magnesite (700° to 1100° C.) at or near the mine; sales of crude magnesite by domestic producers are negligibly small. Careful sorting and even cobbing are necessary at most mines, frequently 2 tons or more being rejected out of 3 tons mined, but mechanical concentration is not attempted. Modern plants, however, include a well designed system of crushing and blending the material to obtain a uniform charge for the kilns. The Northwest Magnesite Co., Chewelah, Wash., adds a small amount of iron (ore, cinder, or scale) before sintering. Dead-burned magnesite is screened through ¾ in. and dust is removed. Caustic magnesite usually is shipped in lump condition, pulverized near the point of consumption (chiefly in roller-type mills), and blended with admixtures for flooring or stucco compositions. Mill capacity is much less than one-half as great as for lime, and power consumption is much greater.

MEERSCHAUM[23]

Meerschaum usually occurs as an alteration product of magnesite or serpentine, or possibly other high-magnesium minerals. In the Turkish deposits (near Eski-Shehir) which have furnished virtually all the world supply, the valuable mineral occurs in nodules, ranging from the size of an egg to that of a football and associated with fragments of magnesian and hornblende rocks, in drift material that has been cemented by lime. In New Mexico, near Sapillo Creek, where mining was done for a few years, the meerschaum occurs in veins, lenses, seams and balls in limestone.

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[22] F. F. Hintze and L. H. Lange: Separation of Cyanite and Mica from Quartz, Feldspar, and Other Gangue Minerals of a Mica Schist. U.S. Bur. Mines *Rept. of Investigations* 3085 (1931).

[23] A. V. Petar: Meerschaum. U.S. Bur. Mines *Inf. Circ.* 6780 (1934).

In Turkey preliminary treatment of the hand-sorted material is confined to the removal of adhering dirt, drying, and rough polishing, but at the New Mexican operation the bulk of the material required mechanical concentration. The latest flow sheet[24] was reported somewhat as follows: From mine to grizzlies to Samson crusher to Blake crusher (all material reduced to 2 in.); elevator to automatic drier to sizing trommel (mesh not given); oversize to coffee mills, undersize to fine-ore bins; "part of fine ore" (larger sizes?) to Krom concentrator, "other as sized" to Wilfley table; all tailings from Krom concentrator treated over Wilfley table with a 98 per cent recovery. The meerschaum, being lighter than the gangue (sp. gr., 1 to 2), was washed down the side of the table, while the waste was carried to what is usually the concentrate end. Early efforts to rely solely upon the Krom concentrator were not successful either as regards recoveries or purity of product, even after an automatic drier was substituted for the sun-drying platform.

MICA[25]

Mica comes from the mine, hand-sorted, in rough crystals or books of various shapes and sizes, but before it is sold to electrical concerns and others that purchase the material for further manufacturing it preferably should be separated into three classes of raw material; namely: (1) sheet, (2) punch, and (3) scrap. Punch mica is mica large enough to yield a circle 1¼ or 1½ in. in diameter; the smallest sheet mica is one that will cut a rectangle 1½ by 2 in. Prices are scaled according to size of sheet and according to quality, whether (1) clear, (2) slightly stained or spotted, or (3) heavily stained or spotted. Mica houses or trimming sheds, where mica is stored and prepared for market, are provided at the largest mines, but small producers and feldspar miners who recover mica as a byproduct sell their output either as it comes from the mine or roughly graded to firms that prepare it for the trade. While only the simplest equipment is employed, the preparation of mica for the market requires good judgment based upon long experience and knowledge of market conditions.

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[24] F. V. Bush: Meerschaum Deposits in New Mexico. *Eng. & Min. Jnl.* (1915) **99**, 941–943.

[25] W. M. Myers: Mica, I.—General Information. U.S. Bur. Mines *Inf. Circ.* 6205 (1929).

T. Antisell: Mica Mining and Milling Methods. *Eng. & Min. Jnl.* (1926) **122**, 894–896.

W. M. Myers: The Use of Flocculating Reagents for the Recovery of Fine Mica. U.S. Bur. Mines *Rept. of Investigations* 2798 (1927).

H. M. Urban: Mica-Mining Methods, Costs and Recoveries at No. 10 and No. 21 Mines of the Spruce Pine Mica Co., Spruce Pine, N.C. U.S. Bur. Mines *Inf. Circ.* 6616 (1932) 15.

The rough books are first cobbed with small hammers and smartly rapped to remove loose dirt and small fragments of mica; badly ruled, fishbone, and otherwise distorted or defective mica that will yield no sheet or punch sizes is rejected at once as scrap. The remaining books are rifted or split into sheets ⅙ in. thick or less, using a stout double-edged knife. Only thoroughly experienced rifters are able to split the books so as to obtain a maximum yield of large-size mica. The smaller sizes, punch mica and washer stock, are commonly left with rough edges and may go direct to the machines for making disks, washers, and other punched forms. Larger pieces are trimmed, using a sharp, thin-bladed knife, which usually is held at an angle so as to produce a beveled edge. This removes the ragged or partly crushed edges and prepares the way for further splitting. Much domestic mica is only rough-trimmed, some of it being only thumb-trimmed, which means that only loose pieces are broken from the edges of the rifted mica, the object being to remove as little as possible of the sound material. On the other hand, most imported micas are close-trimmed to remove all imperfections. In "sickle trimming," irregular shapes may be produced, often with reentrant angles where flaws or cracks were cut out. Imported knife-trimmed mica is in irregular polygons with straight, unbeveled edges, and shear-trimmed mica is in rough rectangles. Trimming, of necessity, produces a large amount of waste, and obviously the processor or manufacturer, after cutting out his pattern, will have left much larger trimmings for cutting up into smaller patterns if he buys rough-trimmed mica than if he buys close-trimmed mica cut from the same block. Many buyers, however, fail to appreciate this fact and therefore pay much higher prices for their raw material.

Owing to the development of new binders and improved methods of manufacture, built-up mica is making inroads on the use of sheet mica. Muscovite splittings are imported mainly from India. Phlogopite splittings are produced in Canada and Madagascar. Splittings are thin leaves or films approximately 0.001 in. thick and are prepared by hand with the aid of a small splitting knife from smaller sizes of mica, up to say 1 by 3 in. Attempts to produce the splittings mechanically, as by bending the books back and forth so that the films will slip on one another, have not been wholly successful, especially with muscovite.

Scrap mica, both mine waste and trimming-shed or shop scrap, is used for making ground mica. Sericite and high-grade biotite schist are ground for similar purposes in a few places, and for certain uses byproduct mica from clay washing is employed. Although mica is considered a soft mineral, it is one of the most difficult to grind. Its cohesion, toughness, flexibility and smooth surfaces make it difficult for any type of mechanical equipment to grasp the particles so that the constituent laminae can be torn apart and reduced in size.

For dry grinding, hammer mills have doubtless been most employed, the ground mica discharged through the mill screens being elevated to multiple-deck vibrating screens that produce the sizes required. Marcy rod mills have been employed with satisfactory results, and in older plants buhr mills and emery mills were used with varying degrees of success. Preliminary heat treatment makes grinding easier but alters the appearance and strength of the flakes. Dry-ground mica is a relatively cheap product and is used largely in coatings for prepared roofings.

Wet grinding is generally preferred for smaller sizes or where a brighter product is desired. Practical men claim that the sheen of mica is due to the fact that it contains "oil" or sap which is lost by weathering, heat, or impact. Home-made equipment is used in many plants, and a great secrecy surrounds the operations. The classic machine is a heavy tub built of wooden blocks in which the charge is abraded by a wooden wheel or disk revolved by a vertical shaft and pressed down upon the charge by a spring above. Whenever possible the endgrain of wood is pointed inwards to resist abrasion. Washed scrap mica is charged with only enough water to provide some lubrication, then the wheel is pressed down until it takes a firm hold on the mica. The friction churns the mass, and the heat is such that the water almost boils. It takes 8 hr. to make a run on 300 or 400 lb. The mill is sluiced out into shallow troughs to settle, after which the water is drawn off, coarse mica is sent back to be reground, and the fine mica is dried on steam tables and bolted, oversize (on 16 mesh) going back to be reground. Edge-runner mills of larger capacity have been introduced at several plants. The pans vary in size up to perhaps 10 ft. in diameter and 3 ft. deep. They are likewise constructed of wood, the bottom being made of endgrain blocks. Four wooden rollers are employed, each 30 in. in diameter and 24 in. wide, arranged so that they can be raised or lowered according to the height of charge in the mill; on clean mica they last about two years. An increasing output of wet-ground mica is produced in pebble mills.

At the byproduct plant of the Harris Clay Co. the sand and mica shoveled from the settling troughs is washed to trommel screens of large diameter (5 ft.), revolving about 30 r.p.m. Undersize contains little mica and is rejected. Oversize is separated on shaking table, the mica, which flows over the riffles, being dewatered and shoveled on a Dutch-oven drier. The dry product is sized on two sets of Rotex screens and bagged.

Mineral Pigments

The principal requirement of a paint pigment is uniformity of color, but fine grain size and low oil absorption are likewise important. In linoleum and oilcloth filler, the chief consideration is frequently price, although low oil absorption is a factor in computing ultimate economy.

For a given type of pigment the relative value is based upon brightness and intensity of color and its strength or staining qualities. The red iron oxide pigments, paint shales, manganese ore, and sundry other materials employed as pigments, either raw or calcined, are prepared for market in a variety of grinding machines. Formerly buhr mills were principally employed, but ball mills and other pulverizers are now used to a large extent. For the softer materials roller mills are indicated. If grinding is done wet, levigation is employed for sizing; for dry pigments air separation is rapidly displacing bolting, which formerly was the universal means of meeting size specifications. Whereas 200-mesh grinding was formerly considered sufficient, a 325-mesh product is now demanded by many consumers, even though the pigment may subsequently be ground in oil for a long period in the paint mill.

Ocher and certain other earthy pigments occur in pockets, seams, or irregular veins in residual or transported clays, sometimes extending downward into cracks and fissures of bedrock. Ordinarily the material as mined must be freed from grit. The Georgia ocher, for example, contains 60 or 70 per cent of quartzite fragments, clay, sand, and a little barite, as it goes to the washer. Usual treatment begins with a log washer, the coarse discharge from which is sprayed over a 12-mesh screen before being sent to waste. The undersize from this screen, after thickening, joins the overflow from the logs, and the slurry then flows slowly through about 150 ft. of wooden launders (10 by 10 in.), sloping about ¼ in. in 16 ft. The sand is shoveled out of these launders continuously by hand. To reduce the large loss of ocher in this sand, James slime separators or classifiers (14-ft. cones) were installed at one plant, the upward current being adjusted to make a separation at 300 mesh. Spigot discharge is scrubbed in a conveyor with double flights forming an integral part of the cone, the washings being pumped back into the cone so that a recovery of 96 to 98 per cent of ocher is obtained.[26] Dorr thickeners are used to produce a pulp with about 60 per cent solids. Another flow sheet includes a Dorr hydroseparator following the log washer; one Dorr classifier scrubs the sand, and a second Dorr classifier in closed circuit with a ball mill grinds the coarse ocher and returns it to the hydroseparator.

Instead of filtering and drying the thickened pulp, Georgia practice is to spatter it on slowly revolving drums, 10 ft. long and 4 ft. in diameter, heated with live steam at 80 lb. pressure. The spattering device is a 3-in. shaft studded with 3-in. bolts or arms rotated in the pulp at 200 r.p.m. The ocher remains in contact with the heated surface only long enough to be dried; other types of driers tend to allow portions of the product to be heated too long, thereby darkening the product or such portions of it as are in contact with the steam pipes or other source of heat.

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[26] A. H. Hubbell: An Improved Way of Washing Ocher. *Eng. & Min. Jnl-Pr.* (1924) **118**, 336.

MONAZITE

Monazite is widely, though sparsely, distributed in granitoid rocks, but commercial deposits are black sands, chiefly beach placers which now are worked for ilmenite with monazite and zircon as byproducts. Natural concentration by wave action (or wind action in dune deposits) generally needs to be supplemented by further washing in sluice boxes or tabling. The mixed concentrates are finally separated by electromagnets, often supplemented by electrostatic treatment.

PHOSPHATE ROCK[27]

The phosphate rock of the Western States and the blue rock of Tennessee are of commercial grade as mined and need only to be crushed and dried. Tennessee brown rock and the hard-rock and land-pebble phosphates of Florida, which contribute more than 90 per cent of the tonnage mined in the United States, occur in residual deposits and must be washed. In the land-pebble fields the matrix of sand and clay carries from 10 to 50 per cent of phosphate in the form of embedded grains and pebbles ranging up to about 1½ in. in diameter. Hard rock occurs as large fragments, boulders, plate rock, and smaller sizes in a matrix with clay, sand, and soft phosphate, the recoverable phosphate seldom exceeding 25 per cent of the total.

From mine cars the matrix is generally dumped on a roll or bar grizzly with appropriate spaces (4 to 10 in.), phosphate rock being sledged through and waste rejected. Sometimes a roll crusher is employed for reducing oversize, the product being all put through a trommel with holes 4 in. in diameter or less, according to the limiting size adopted. In the Florida land pebble field, where the rock is smaller and mining is done by hydraulicking, the matrix, already disintegrated by the monitors, is picked up by the sand pumps from the mine sumps and spouted at the mill on to a flat screen followed by a trommel which separates out clay balls, limestone boulders and debris, the limiting size usually being 1½ in. A stationary screen with $\frac{3}{32}$ to $\frac{3}{64}$-in. slots may follow or precede the limiting screen. The undersize is waste, and the oversize goes to log washers, the overflow from which is also waste. The discharge from the log washer is screened and if necessary passed through a second log washer. The latter, however, may be replaced by a cylindrical scrubber, wash trommel, emulsifier, or some special machine; the Hardinge scrubber in particular seems to have rendered a good account of itself in elutriating residual clay balls. Thenceforward the washed solids may be merely sized in trommels or on vibrating screens. The coarse sizes, over about ⅜ in., are often hand-picked, while intermediate sizes down to about $\frac{1}{16}$ in. are sent separately to storage bins.

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[27] J. A. Barr: The Development and Application of Phosphate Flotation. *Ind. & Eng. Chem.* (1934) **26**, 811–815.

C. E. Heinrichs: Economic Results of the New Technique in Phosphate Recovery. *Min. & Met.* (1933) **14**, 329–32 and 350.

J. B. Huttl: Mining and Processing Hard-Rock Phosphate. *Eng. & Min. Jnl.* (1932) **133**, 336.

N. K. Karchmer: Flotation of Apatite in Russia. *Eng. & Min. Jnl.* (1932) **133**, 429–32. (Oleic acid and peat tar are used as flotation agents.)

H. M. Lawrence, F. D. DeVaney, R. G. O'Meara and others: U.S. Bur. Mines *Repts. of Investigations* 2860, 2925, 3018, 3023, 3105, 3139 and 3195.

H. S. Martin: Milling Methods and Costs at No. 2 Concentrator of the Phosphate Recovery Corporation. See p. 466, this volume.

Progress in Phosphate-Rock Industry is Reflected in New Florida Plant. *Pit & Quarry* (1931) **23**, 19–25.

R. W. Smith: Mining and Washing Phosphate Rock in Tennessee. *Eng. & Min. Jnl.* (1923) **115**, 221–226.

J. K. Towers: Improvements in Mineral Scrubbing. *Eng. & Min. Jnl.* (1932) **133**, 174.

W. H. Waggaman and H. W. Easterwood: Phosphoric Acid, Phosphates, and Phosphatic Fertilizers. Amer. Chem. Soc. *Mon.* 34 (1927) Chem. Cat. Co.

In the Tennessee washers, sand (under about 8 mesh) has generally been treated in dewatering cones and tables have been employed, the imperfect separation being based upon the fact that quartz grains are smaller and so ride higher on the table. In a typical land-pebble mill, however, everything under about 1.5 mm. had to be thrown away; it contained 12 to 45 per cent bone phosphate of lime, but there was no way to recover the phosphate grains when they approached the sand grains in size, as the specific gravities are virtually the same. By treating such material by flotation, however, recoveries at most deposits can be doubled, and the concentrates so produced will grade up to 3 per cent higher even than the regular washer product. In Tennessee, where most of the high-grade deposits are exhausted, operators have barely been able to meet market requirements, but by employing flotation a brown-rock plant at Wales, Tenn., boosted the B. P. L. content of its product to between 75 and 80 per cent and reduced the iron and alumina to below 3 per cent, compared with 70 to 75 per cent B. P. L. and 5 to 5.5 per cent iron and alumina by former methods. Flotation feed is classified at 28 to 35 mesh, oversize being ground in a rod mill in closed circuit with the classifier. The reagents are fatty acids or soaps in combination with a suitable collector in a slightly alkaline circuit. Minerals Separation cells are preferred both for roughing and cleaning.

Flotation concentrate is dewatered and elevated to silos with filter panels where it drains to 13 per cent moisture. All concentrates and washed rock must be dried. Accepted practice for some years was to use 48-in. by 30-ft. directfired, counter-current rotary kilns, but better economy has been obtained by using a 72-in. by 64-ft. direct-fired, parallel-current kiln.

Selective oiling and tabling (agglomerating) is employed at one or more Florida land-pebble plants and, being applicable to sand sizes, is a logical installation where the size of deposit (or debris pond) is small or where other factors do not justify the extra cost of a fine-grinding plant to prepare the sludge for flotation. The phosphate grains (sp. gr., 2.85) after oiling with fuel oil and soap (sodium oleate) are discharged rapidly over the side of the table and unoiled sand (sp. gr., 2.65) at the end of the table.

For making superphosphate, the rock must be ground to a fineness of about 90 per cent through 80 mesh and 45 per cent through 200 mesh. The material is easy to grind in air-swept Hardinge mills. Dry-grinding units were formerly used exclusively, but some wet grinding is now being done.

PYROPHYLLITE

North Carolina deposits of pyrophyllite are high grade, and no mechanical concentration is required. Ground pyrophyllite is sold as "talc," which it resembles in physical properties.

SALINES[28]

As common salt, boron minerals, and various other salts of soda, potash, magnesium and calcium are soluble in water or can readily be converted into soluble salts, they are commonly recovered by leaching followed by chemical treatment, evaporation, and crystallization—processes that are primarily in the field of industrial chemistry. Processes have been developed, however, that are purely mechanical, using ordinary ore-dressing equipment, but instead of pure water the mill circuit employs a saturated solution of the salts desired. The principle has been employed commercially for the removal of clay (in log washers, etc.) but may have applications in table treatment of complex salts of different density provided the individual crystals can be separated readily. The Bureau of Mines has separated sylvite and halite successfully in its laboratories at Rolla, Mo. Borax is now obtained mainly from rasorite (or kernite), but when colemanite was the principal source it was concentrated by mechanical means before being sent to the refinery. Calcining the run of mine product crushed through ¾ in. in rotary calcining kilns kept at 800° C. resulted in decrepitation of the colemanite, which then was separated on 24-mesh shaking screens; clay was removed by pneumatic tables or air classifiers. The oversize, comprising ulexite and gangue (shale, limestone, etc.), was ground to pass 25 mesh and wet-concentrated by Harz jigs and tables; calcined ulexite is only slightly soluble in cold water and is somewhat lighter than the gangue. In preparing *rock salt* for market a more or less complete series of crushing and screening operations is required, generally with one or more picking belts and magnetic pulleys to remove impurities, including tramp iron and detonator wires.[29]

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[28] G. R. Robertson: The Trona Enterprise. *Ind. & Eng. Chem.* (1929) **21**, 520–524.

SAND AND GRAVEL[30]

Development of a successful sand and gravel enterprise is based upon the local market. Main outlets are for use as : (1) concrete aggregate, (2) road material, (3) railroad ballast and (4) building sand. Specifications covering sizes and purity of these products vary in different localities. Special sands—such as molding sand, glass sand, filter sand, abrasive sands, and engine sand—must conform to still more rigid specifications, but as even they are low-priced commodities treatment costs must be kept so low as to eliminate the possibility of extensive beneficiation. Broadly speaking, the nature of the product is dictated mainly by the nature of the material in the bank. Nevertheless, few deposits yield material sufficiently pure and uniform to be shipped without some treatment except for low-grade uses; at practically all large operations the product must at least be sized, and consumers are insisting more and more that it should also be washed.

Plant practice naturally varies widely. The simplest treatment, of course, is hand screening in the pit, and next to that is a single revolving screen with several sections for sizing dry material. Even when washing is a secondary consideration, however, wet screening doubles plant capacity, and it then becomes a rather simple matter to add washing jets and possibly a scrubber section on the trommel as a further means of eliminating soft clay or loam. Film clay has to be scrubbed off, and tough clay calls for more drastic treatment unless the clay balls can be hand picked. For removing vegetable trash, a washing cylinder or other stirring device with suitable skimming arrangements may be required. Jigs may be employed for removing lignite, coal, and other light impurities and for giving a further scrubbing. "Sand rock" and slate are undesirable, and shale, particularly varieties that swell when wetted, may render the product unsalable. Soft stone can occasionally be removed by jigging, but the more efficient eliminators work on the principle of crushing or grinding it small enough for it to be washed away. Mica may be removed from sand to some extent by classification, and soluble salts are removed automatically by any process employing quantities of fairly pure water.

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[29] H. D. Keiser: Processing Rock Salt for Market. *Eng. & Min. Jnl.* (1932) **130,** 63–66.

[30] C. R. Fettke: American Glass Sands, Their Properties and Preparation. *Trans.* A.I.M.E. (1926) **73,** 398–423.

E. Shaw: Washing and Sizing Sand and Gravel. Trans. A.I.M.E. (1926) **73,** 424–433.

J. R. Thoenen: Economics of New Sand and Gravel Deposits. U.S. Bur. Mines *Econ. Paper* 7 (1929). Contains bibliography.

W. M. Weigel: Preparation and Use of Industrial Special Sands. *Trans.* A.I.M.E. (1926) **73,** 434–448.

F. M. Welch: Design of Sand and Gravel Washing and Screening Plants. *Rock Products,* **134.** A series of eight articles, April to November, 1929.

Rock crushers are found at most plants for breaking up boulders and cobbles removed on a scalping screen and sometimes for increasing the yield of small gravel and sand. Some commercial glass sands are sufficiently consolidated to require crushing and grinding (usually in chaser mills). Uniformity of grain size is important for glassmaking as well as for certain other uses. Chemical as well as magnetic treatment has been advocated for reducing the iron content of glass sands, but generally the selling price limits the purification to washing or screening out the fine material, which ordinarily carries the bulk of the iron and alumina. Shaking tables, however, have been employed to take out black sands, pyrite, and rutile.

Screw washers, rake classifiers, drag classifiers, and even log washers may be employed where the proportion of fine sand is high and for silt and clay removal, but surface-current classifiers—either ordinary settling tanks or cones without rising water—are chiefly employed for grading and dewatering sand. Special sands may have to be sized on vibrating screens after drying.

STONE[31]

Quarry methods control to quite an extent the proportion of high-grade finished products secured, and great progress has been made in recent years in designing quarry and mill equipment to reduce waste. Channeling machines making straight cuts 1½ to 4 in. wide were a great advance over blasting, which was used widely in early days, but they too were wasteful and tend to be replaced by wire saws, particularly in slate quarries. The wire makes a cut only about ¼ in. wide and not only wastes little rock in the form of cuttings but also eliminates the "stunning" effect or propagation of fractures by channel bars which sometimes destroys a zone 1 or 2 ft. wide on each side of the cut.

In *limestone, sandstone,* and *marble* mills quarry blocks are cut into slabs chiefly by the gang saw, a series of soft iron blades set in a reciprocating frame and fed with sand or steel shot as abrasive. Subsequent cuts are made chiefly with diamond-toothed or carborundum-toothed circular saws. Surfaces are smoothed and slabs reduced to desired thicknesses with planers operating in much the same way as planers in machine shops. When a smoother surface is desired the slab or block is placed on a rubbing bed (a cast-iron disk 12 to 16 ft. in diameter rotating in a horizontal plane), a barrier holds it in place, and sand and water are applied to the surface of the disk. Carborundum machines are widely used for cutting cornices and moldings, beveling edges and making spindles. Hand cutting and carving are necessary for many architectural units, but pneumatic tools have replaced hand tools and wooden mallets to a great extent.

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[31] Contributed by Oliver Bowles, Supervising Engineer, Building Materials Section, U.S. Bureau of Mines. For further details, see The Stone Industry. New York, 934. McGraw-Hill Book Co.

Granite, which is much harder than other varieties of dimension stone, is milled differently in some respects. Until recent years blocks were subdivided almost universally by drilling and wedging. The surfaces were then worked to an even plane by means of surfacing machines consisting of powerful pneumatic hammers that when guided over the surface chipped off projections by repeated blows. In many modern granite mills both gang saws and circular saws with steel shot as abrasive are used successfully. They conserve material and provide relatively smooth surfaces that are advantageous for following processes. Rubbing beds are not used in granite mills. A smooth finish is obtained with a buffing machine consisting of a power-driven rotary head, which, when guided over the surface, wears it down with successively finer grades of steel shot, carborundum grit or emery. The final polish on both granite and marble is obtained with a buffer head supplied with "putty powder," an extremely fine-grained tin oxide. Carborundum machines are used for cutting shallow grooves and contours in granite blocks, but the wheels must be guided carefully and supplied with an abundance of water.

Sand-blast carving has developed remarkably, particularly in granite. The process consists of covering the surface to be lettered or carved with a rubberlike "dope," cutting the design through the dope to the rock surface with a fine scalpel, and directing a sand blast against the surface to reproduce the design on the stone.

Slate milling[32] differs widely from other types of stone milling. Slate possesses the peculiar property of slaty cleavage, a fissility which permits it to be split into thin, uniform slabs. For roofing-slate manufacture the blocks are split with chisel and mallet until slabs 3/16 or 1/4 in. thick are obtained. They are trimmed to standard sizes with a heavy blade operated with a foot treadle or with a power-driven rotary trimmer that embodies the shearing action of a lawnmower. Slate for the manufacture of blackboards, electrical panels and structural products first is split to the approximate thickness desired and then smoothed and reduced to uniform size by planers, rubbing beds, and surfacing machines similar to those used in marble mills. Carborundum machines generally are used for coping (trimming edges of slabs and cutting them into strips).

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[32] O. Bowles: The Technology of Slate. U.S. Bur. Mines *Bull.* 218 (1922).

The manufacture of *granules* for surfacing prepared roofing is partly a branch of the slate industry, but other materials such as trap rock, rhyolite and silica also are used. The object of the milling process is to prepare a clean product ranging from 8 to 35 mesh in size and to make as small a proportion of fines as possible. Fines are marketed less easily than granules. Quarry rock is first crushed in a gyratory or roll crusher, further reduced chiefly by rolls, and sized with vibrating screens. Fines are rigidly excluded. Raw materials are carefully selected as to color, but granules also are colored artificially, and the synthetic colors are gaining in popularity.

Even in the best regulated mills and quarries there still is considerable unavoidable waste, and means of utilizing it profitably are constantly sought. Waste marble is pulverized and sold as whiting substitute, waste limestone is ground for agricultural use, and waste sandstone is ground and screened for use as building and furnace sand. Other byproducts are ashlar and rough building stone, chicken grit, roofing pebbles, asphalt filler and furnace flux.

The processes of quarrying stone for aggregate, road material, railway ballast, and furnace flux are quite different from those of quarrying dimension stone. Instead of exercising care to procure large rectangular masses free from cracks and flaws, quarry method is designed to obtain a maximum of rather small fragments. Large-scale methods may be followed, the rock usually being broken from the parent ledge by heavy blasts in deep churn-drill holes.

The principal processes involved in milling crushed stone are crushing, elevating, conveying, screening and washing. The objects of the processes are primarily reduction in size of fragments and classification according to size; nevertheless, the element of concentration enters to some extent. Fines are removed and either discarded as waste or sold as byproducts. Clay and other impurities are sometimes removed by washing as the stone fragments pass over screens.

The principal types of equipment for crushing are gyratory and jaw crushers, single rolls, double rolls, cone crushers and disk crushers. Screening equipment has changed during recent years. For coarse screening, trommels have given place to some extent to the disk type, such as the cataract grizzly and the multiroll sizer. Vibrating screens are used increasingly for the finer sizes.

SULFUR AND PYRITE

Sulfur is produced in the United States by the Frasch process, the hot sulfur on reaching the surface being spouted into the center of large bins, where it solidifies. These huge blocks are drilled and blasted, the sulfur (99.5+ per cent pure) being loaded by steam shovel. Commercial flour sulfur (American) is run of mine material ground 95 per cent through 100 mesh. In Sicily, where the crude ore contains 20 to 25 per cent S, sulfur is extracted by fusion and liquation, and refining is done in retorts. Flotation of native sulfur is practicable; Nevada operations employing flotation, as well as a "coalescing method" in hot water, have been described.[33]

Some pyrite has been produced in the United States as a byproduct of coal mining but most is a primary mine product. Methods of concentration are essentially similar to those for other metallic sulfides, but recent advances in differential flotation in Tennessee and elsewhere have released a quantity of pyrite concentrates formerly smelted with associated copper minerals. Lump and fines are marketed separately. Impurities (zinc, lead, arsenic, etc.) that volatilize in roasting are objectionable; copper, gold, and silver, if present in substantial amount, may be paid for (but at a discount).

TALC[34]

Crude talc is worth from $3 to $50 or more a ton. High-grade massive crude talc suitable for crayons and pencils or the special grade employed for "lava" products is carefully selected and cleaned at the mine, large lumps frequently being trimmed with axes and sawed like cordwood so as to facilitate cutting out hard particles and fibrous material. Sometimes it is further manufactured by sawing or carving at the mine, into products worth up to $300 or more a ton. From a tonnage standpoint, however, 95 per cent of the talc business is in ground talc, most of which is produced at the mines. The hard or "fibrous" talc, grading into tremolite, produced in New York State, is ground mainly in tube mills; the tendency is for air separators to replace screens formerly used to eliminate coarse waste from the product. Elsewhere Hardinge mills have been used alternatively with emery mills and roller mills followed by air separators. Where sale is found for a relatively coarse granular product (roofing trade) or where grit and other hard impurities are to be eliminated, suitable pulverizers with a throw-out device have an important application. Ordinary flour mills were employed originally for grinding talc but, despite the softness of the material, were not economical; the buhr mills had little capacity, and silk bolting cloth wore out rapidly. Wet grinding has been proposed for pyrophyllite but apparently has not been commercially adopted for talcose minerals in the United States. Flotation tests[35] on Canadian talc resulted in reducing lime content from 5.7 to below 0.5 per cent, but European buyers contended that the product did not possess the slip qualities of either Italian or Spanish talcs. In grinding talc mixed with harder minerals, each grinding machine and various adjustments of the same machine may have a characteristic selective action. At one plant, for example, the best product (as regards whiteness, slip and fineness) is obtained from a modified vertical emery mill with hard steel disks, but the yield was small.

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[33] H. L. Hazen: Recovering Sulphur. *Eng. & Min. Jnl.* (1929) **127,** 830–831.

H. L. Hazen: Recovery of Sulphur from Surface Deposit. *Min. Jnl.* (Ariz.) (1930) **13,** 7.

[34] R. B. Ladoo: Talc and Soapstone—Their Mining, Milling, Products, and Uses. U.S. Bur. Mines *Bull.* 213 (1923).

For grinding soapstone waste,[36] a hammer mill is used for secondary crushing (from about 1 to ¼ in.) and a ball mill for fine grinding. Coarser products (main tonnage minus 40 plus 48 mesh) are sized on Hum-mer screens, but the tube-mill product is air classified to 97 per cent through 300 mesh.

VERMICULITE[37]

At Libby, Mont., the material broken from the face is screened at the mine, fines being rejected. At the mill, the oversize is dried to under 3 per cent moisture in a rotary kiln (heated to only 180° F. to avoid exfoliating at this stage), passed through a hammer mill, and then graded into three sizes, under ½ in. plus ¼ in., under ¼ in. plus 10 mesh, under 10 plus 28 mesh (plaster size), and fines, all under 28 mesh being handled as one grade. The grinding and screening eliminates much foreign material, but a final cleaning occurs in the Schundler process. Originally a rotary kiln heated to 800° F. was employed for expanding Zonolite, but better results are obtained by dropping a measured hopperful of the material vertically through one battery of gas burners and then down the inclined (30°) bottom of a Schundler expanding furnace, where it is met by three or four other batteries of burners at intervals of 6 ft. After being heated to about 1800° F. for 4 or 5 sec. the ore reaches the discharge end of the furnace, where it is picked up by a current of air, thereby classifying the product. Finished material is carried to the loading hoppers, leaving behind any insufficiently expanded Zonolite as well as impurities.

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[35] R. K. Carnochan and R. A. Rogers: Canada Dept. Mines, Mines Branch, *Rept. of Investigations* (for 1932), Nonmetallic Min. Sec. 736 (1934) 231–4.

[36] H. H. Hughes: Soapstone. U.S. Bur. Mines *Inf. Circ.* 6563 (1932), 13–15.

[37] A. V. Petar: Vermiculite. U.S. Bur. Mines *Inf. Circ.* 6720 (1933), 7–8.

W. S. Steele: Vermiculite—Production and Marketing by the Zonolite Company. *Trans.* A.I.M.E. (1934) **109**, 418.

Iron-ore Beneficiation in the Lake Superior District

By E. W. Davis,* Member A.I.M.E.

Iron-ore beneficiation methods are determined largely by blast-furnace requirements. There are still many millions of tons of direct furnace ore in the Lake Superior district and blast furnaces are designed and operated for the most efficient use of this material. Beneficiated ore must be so prepared that existing furnaces may use it without alteration in design or radical changes in practice. Many attempts have been made to develop new furnaces or processes for smelting iron-ore concentrate not suitable for blast-furnace use, but these processes have gained little importance. It is, therefore, the endeavor in beneficiating iron ore to make a product that will give at least as good results as direct furnace ore in existing blast furnaces. The term "beneficiation," as applied to iron ore, does not necessarily mean concentration, but more accurately means "dressing up" the ore so that it is more acceptable to the blast-furnace operators.

In modern blast-furnace practice the structure of the ore is quite as important as the chemical analysis. Certainly, the higher the iron content and the lower the silica, phosphorus and sulfur, the more desirable the ore becomes; but if the structure is bad, that is, if the blast-furnace gases can penetrate the charge only with difficulty, the ore is undesirable regardless of its analysis. This means that the ore should be as uniform in size as possible and free from fines, and also that the individual particles of ore should be pervious to the reducing gases of the furnace. Structure and purity are the primary requirements for a furnace ore and structure is just as important as purity.

Iron-ore beneficiation processes can be divided, therefore, roughly into two main classifications: (1) processes that primarily improve the structure, and (2) processes that primarily improve the chemical analysis. During the past 10 years approximately 25 per cent of the ore shipped from the Lake Superior district has been beneficiated. Of this about half has been crushed and screened, a simple operation that primarily improves the structure but in some cases also improves the analysis slightly. The other half of the shipment of beneficiated ore, amounting to an average

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of about five million tons per year, is concentrate; that is, the analysis has been improved materially.

Concentration operations, of course, are more complicated, and in the future will certainly be much more important than the crushing and screening operations. The amount of low-grade ore more or less amenable to concentration in the Lake Superior district is enormous. It constitutes a potential ore reserve many times greater than the tonnage of direct furnace ore. It is with this type of material that the engineers and metallurgists are more concerned. No general statement can be made regarding the nature of the low-grade ores of the Lake Superior district. Radical variations occur in the structure and nature of the iron-bearing mineral in the different districts and in different parts of the same district. The iron-bearing minerals are largely limonite, hematite and magnetite, but specular hematite, pyrolusite, pyrite and ilmenite are also abundant. Structurally this material varies from soft, painty ore to hard, dense rock. The minerals occur as free grains or masses of various sizes, fine intergrowths often of several iron-bearing minerals together, bands of various widths and purity, and as conglomerate masses. About the only generalization that can be made is that the predominant gangue mineral is usually silica. The iron content of the low-grade ores varies from 20 to 50 per cent, probably averaging around 30 per cent. Some of the material, however, contains as much as 60 per cent iron, but cannot be utilized without beneficiation on account of its structure or chemical composition. The concentration engineer, therefore, has a wide choice of raw materials to choose from, but since the tonnage available is so great compared to the demand, and since the concentrate sells for only about $3 per ton at the mine, successful applications of the various beneficiation processes have been made only where the simpler methods can be used and where mining and other costs are the lowest.

Crushing and Screening

The crushing and screening operation is the simplest of the beneficiation processes, producing annually several million tons of furnace ore. Plants of this kind are found on nearly all of the iron ranges of the Lake Superior district. The usual procedure is to deposit the ore on large movable or stationary grizzlies, the oversize sometimes going to waste and sometimes being crushed, after which it joins the grizzly undersize. The shipping ore is usually all finer than 6 in. and is, therefore, much more acceptable to the blast-furnace operators than the original material containing 2-ft. or 3-ft. lumps.

Washing

The Mesabi Range, which contains the largest deposits of low-grade ore in the Lake Superior district, produces most of the beneficiated ore, for two important reasons: (1) The ore deposits lie flat and mining costs are low; and (2) much low-grade material is found associated with the direct furnace ore and it is often necessary to move the low-grade material in order to mine the direct ore. Most of the concentrate produced on the Mesabi Range is made in washing plants. Wash ore is distinguished by the fact that the fine material is low-grade and the coarse lumps are high-grade. It is only necessary to wash and, to a certain extent, scrub the fine sand away from the coarse material to produce a merchantable concentrate. The flow sheet of a modern washing plant includes crushers, washing trommels and log washers or Dorr washers, and bowl classifiers. The concentrate is high-grade, quite free of fines, and is a very desirable furnace product. Normally, about five million tons of wash-ore concentrate is produced in the Lake Superior district each year, practically all of which comes from the Mesabi Range.

Jigging

Jigging plants operating on the Mesabi Range produce several hundred thousand tons of concentrate annually. The jig ore differs from the wash ore in that the silica has not been broken down into fine sand but is still consolidated, and crushing to 1 in. or finer is necessary in order to free the particles of iron oxide and gangue. After crushing, the ore consists of four distinct varieties of material, and the proportions in which they exist determine whether or not jigging will be satisfactory. These four constituents are dense hematite, porous hematite, dense silica and porous silica. It is a simple matter to recover by jigging the dense hematite and reject the porous silica, but a separation of the porous hematite from the dense silica is very difficult. Therefore, for a good jig ore the proportion of porous hematite and dense silica should be small, as these will go into a middling product that is too poor to be included in the concentrate and too good to be included in the tailing. At the plants now in operation large stockpiles of this lean material are accumulating. In these plants the flow sheet consists of crushing to minus 1½ in., washing for the removal of low-grade fines, screening to several sizes, and jigging each size separately. The concentrate produced is high-grade and quite free of fines, making a desirable furnace product.

Magnetic Concentration

A number of years ago a large magnetic-concentration plant was built at the eastern end of the Mesabi Range, where the ore is very hard and dense and in which the iron oxide exists as magnetite. The ore is slightly banded, but the bands are neither clean magnetite nor gangue. It is necessary to crush this ore to minus 150 mesh before a high-grade concentrate can be secured. The flow sheet used in this plant was crushing to minus 6 mesh and cobbing out about one-third of the ore as tailing.

The remaining two-thirds was crushed to minus 150 mesh and concentrated in magnetic log washers, resulting in an over-all ratio of concentration of about three tons into one. It was then necessary to dewater and sinter the fine concentrate. The sintered product was high-grade, low-phosphorus ore of very desirable structure, but shortly after the plant was put in operation a decrease in ore prices made operations unprofitable. This plant demonstrated the possibility of producing high-grade ore from the low-grade magnetite-bearing rock that exists in very large quantities toward the eastern end of the Mesabi Range.

Drying and Sintering

Cuyuna Range ores tend to be fine in structure and high in moisture and ignition loss. To counteract this difficulty, drying and sintering plants are used. The drying plants consist of coal-fired rotary driers which reduce the moisture to about 10 per cent. A further reduction in the moisture is usually not desirable on account of the dusty nature of the product. In the sintering plants all of the free and combined water contained in the ore is removed and a high-grade material of good structure is produced. The Cuyuna ores usually contain manganese, which increases their value.

In Michigan and Wisconsin, crushing and screening constitute the principal beneficiation process. A number of attempts have been made in the past to concentrate the lower grade ores but up to the present time little concentrated ore has been shipped. Intensive research work is being carried on at the Michigan College of Mines, and it is expected that concentration methods will be developed that will make possible the utilization of some of the leaner ore on the Gogebic, Menominee and Marquette Ranges.

Recent Developments

During the past few years a number of new developments have appeared in connection with the beneficiation of Lake Superior ores. It has become evident to several of the mining companies that the good wash ore that has furnished the bulk of material for the washing plants is disappearing. There still remain in these properties from which the wash ore is being removed large tonnages of low-grade material that does not wash up to grade. It has been shown that it is possible by jigging this material or certain sizes of the material to remove sufficient silica to produce a satisfactory concentrate. For this reason, jigging operations of a more or less experimental nature have been carried on in connection with some existing plants. Different types of jigs have been used and the indications are that the future will see considerably more jigging on the iron ranges either at existing washing plants or as independent jig plants.

The recovery of fine, free particles of iron oxide in washing-plant tailing has been a problem since the first washing plant was constructed. Originally tables were used but these were replaced later by bowl classifiers, which required much less floor space and operating attention. At the present time the Hydrotator and the Rheolaveur separator are being used to a limited extent in Mesabi Range plants. The fine ore that these machines recover can usually be absorbed in the coarser concentrate without seriously affecting the value of the ore. If any considerable amount of this fine material were produced, however, such as would be the case if old tailing ponds were reworked, sintering would undoubtedly be required.

A new plant has been put into operation this summer on the Mesabi Range for the roasting and magnetic concentration of jig tailing. This minus ¾-in. tailing is roasted in a shaft-type magnetic roasting furnace and is then concentrated on magnetic concentrating machines. This method of concentration has been experimented with for many years but this is the first attempt to apply this process commercially on the Mesabi Range. If this method of concentration proves to be economically successful, many millions of tons of lean ore now in stockpile may be concentrated.

Occasionally small quantities of wash ore are encountered in open pits from which the direct furnace ore is being removed. If the amount is not sufficient to warrant the erection of a washing plant, this low-grade material is left in the mine or in stockpiles. Recently a portable washing plant has been assembled that can be moved about on a railroad track. This unit can be moved about in the pit and the ore loaded directly into the plant. By use of this equipment, concentrate can be produced economically from small deposits of wash ore that otherwise could not be handled.

Future Trends in Iron-ore Beneficiation

The future of the Lake Superior district will depend largely upon the extent to which the low-grade ores are utilized. There is perhaps a 35-year supply of direct furnace ore still available. This material will be mined, of course, but whether or not the Lake Superior iron-ore industry and the iron and steel industry depending upon it will continue to be an important factor will be determined largely by the extent to which the low-grade ores of the Lake Superior district are utilized. Many things other than the technical and engineering difficulties involved will affect the establishment of a large low-grade iron-ore industry. Taxation is an important consideration, especially in Minnesota. If the ad valorem system of taxation now in use is not altered or abandoned with regard to the low-grade ores, the extensive utilization of this material is impossible. It is difficult to tell just what effect the St. Lawrence Deep

Waterway will have on the iron and steel industry. It is unlikely that new markets will be developed on account of the waterway for Lake Superior ores, but it is probable that foreign ore will be brought in greater abundance to the Lake furnaces. Therefore it seems that this waterway will to some extent decrease the requirements for Lake Superior ores, which will, of course, postpone the necessity for utilizing the low-grade materials. The increased use of iron and steel for scrap is reducing the demand for iron ore. In addition to this, the use of alloys has made possible a decrease in the weight of load-carrying members and has made steel plates corrosion-proof, thus increasing their life and eventually bringing them back into the scrap market. New uses for large tonnages of iron and steel have not developed recently. The most encouraging field seems to be the use of cast iron for pavement surfacing and foundation plates. This has developed into a large industry in England and may develop into a still larger one here. New uses for iron and steel will have a marked and immediate effect on the attitude of the iron-ore producers toward the low-grade ores. The concentrate that will be produced from most of the low-grade ore will be too fine for blast-furnace use, and it will be necessary to agglomerate this concentrate in order to produce a satisfactory furnace ore. If it is possible to change the smelting furnace to suit the fine ore rather than to change the ore to suit the smelting furnace, the utilization of the low-grade ores will be greatly stimulated. This very thing was done in the copper industry and there is no reason to believe that the modern blast furnace is the ultimate in iron-ore smelting. All of these considerations, most of which are outside the province of the ore-dressing engineer, have a direct bearing on the beneficiation of low-grade ores, and much consideration must be given to them before large investments can be made for the construction of beneficiation plants.

Mining engineers and metallurgists are working on the more technical phases of the problem, improving present practice and developing new processes. Useful work along this line is being conducted in at least four university laboratories and by a number of independent engineers and engineering organizations. The direction that these investigations are taking indicates just where the need is greatest and where new developments are to be expected in the future.

Jigs will certainly be used much more extensively in the future than at the present and several new types of jigs are being developed for the treatment of iron ore. Iron-ore jigs must be designed to produce a large quantity of concentrate and a comparatively small quantity of tailing. The present tendency is to develop a rather long and narrow single-compartment jig along which the ore moves rapidly, the heavier portions being drawn off at a number of points throughout the length of the jig bed and the tailing overflowing at the lower end. In the first few draw-offs concentrate is secured, and middling is produced further along the jig. This middling is crushed to the required extent and returned to the jig feed. The jig, therefore, operates on unsized material, although with many ores washing of the feed is desirable for the elimination of low-grade sand and slime. The necessity for the use of such auxiliary apparatus as dewaterers, elevators, pumps and crushers is one of the main problems in connection with iron-ore jigging where tonnages are large and operating costs must be very low.

Tabling disappeared from the iron-ore beneficiation plants about 15 years ago, but there is some indication that it may again be found useful. New types of riffles and head motions have been developed and considerable experimental work has been done in connection with the tabling of iron ore as coarse as ½ in. Extensive experimentation has also indicated the possibility of the concentration of the Cuyuna manganiferous ores. New ideas are being developed in connection with table design and operation, which may make these machines more useful to the iron-ore producer.

For many years the magnetic roasting of iron ore has been experimented with. The chief difficulty has been low magnetic roasting efficiency with high fuel consumption. Recently a shaft type of roasting furnace has been installed near Nashwauk, Minn., which may develop into a satisfactory furnace for roasting iron ore. The number of heat units actually consumed in converting hematite to magnetite is small, but it is necessary to bring the ore to a red heat before reduction takes place rapidly and it is then necessary to cool the ore before allowing air to come into contact with it. In this new furnace the hot roasted ore is cooled in a water bath and most of the heat contained in the red hot ore is recovered as steam. By utilizing the steam, a considerable proportion of the heat consumed in the process can be credited back to the furnace. Fuel oil is used for combustion and for reduction in this furnace. If a cheaper fuel, such as natural gas, can be secured, large quantities of low-grade ore and rock now considered worthless can be concentrated cheaply.

Considerable experimental work has been done on the flotation of iron oxide. In addition to the difficulty of floating the oxide minerals, the iron usually exists as different oxides and with different degrees of hydration in the same sample. In the manganese ores the iron and manganese oxides are often so closely interwoven that the finest of grinding separates them only imperfectly. However, so much has been accomplished in late years with the flotation process that new developments may make possible the commercial concentration of iron ore by this method. This process would be especially useful in the recovery of fine mineral from the rejects from other concentration operations.

Beneficiation of iron ore by leaching has received little consideration except for the removal of small amounts of harmful elements, such as sulfur and phosphorus. For the recovery of manganese from the Cuyuna ores, however, the leaching process has been carefully worked out. The Bradley leaching process for manganese recovery consists in roasting the ore to convert all of the manganese to manganese monoxide. This oxide is then leached out with ammonium sulfide and then precipitated as a manganese hydroxide by ammonia gas. This process forms a completely closed cycle, producing very pure manganese hydroxide and iron oxide than can be concentrated magnetically. Both products require sintering before they can be used in blast furnaces. The process was perfected on Cuyuna ore, which contains about 15 per cent manganese and 25 per cent iron. This process makes possible the production of very high-grade manganese ore from the low-grade Cuyuna deposits, which contain immense tonnages of this material. This process has not been put into commercial operation but is available when prices and demands warrant its use.

Mention has been made of the fact that a large proportion of the concentrate that eventually will be produced from the Lake Superior low-grade ores will be too fine in structure for use in existing blast furnaces, and either the concentrate must all be agglomerated or a new type of smelting furnace must be developed. Considerable experimental work has been done in the development of a reverberatory smelting process for iron ore similar to the reverberatory smelting process used for copper ores. Surprisingly encouraging results have been secured. The fine concentrate mixed with coal dust is fed in along the side walls of the reverberatory furnace and the charge melted down much the same as with the copper ores. The slag and metal are tapped off separately and waste-heat boilers recover the heat loss in the furnace gases. It is proposed to generate electric power from this waste heat, which will be used in electric furnaces to convert the furnace metal into steel. This process has not been put into commercial operation but several hundred tons of metal have been made, some of which has been converted into steel and rolled into very satisfactory bars. The commercial development of this process will make possible the utilization of the fine ores which, at the present time, must be agglomerated.

The production of sponge iron from fine concentrate has also received much attention. Several attempts have been made to produce sponge iron commercially in this country but without great success up to the present time. The development of the new high-temperature alloys and better refractories will help to make this process more satisfactory, and the future will undoubtedly see methods perfected for economically producing sponge iron from fine, high-grade concentrate.

All of the concentration plants now in operation work only during the summer months, which constitute the shipping season on the Great Lakes. Underground mines often operate the year around, stockpiling the ore during the winter months. There is no necessity for operating the open-pit mines during the winter on direct furnace ore, but there would be some advantage in operating concentration plants the year around. There is no serious obstacle to mining the ore and concentrating it during the winter, but it has been found that large stockpiles of wet ore made during the winter will not thaw out during the following summer. Several ideas have been suggested for overcoming this difficulty and undoubtedly, in the future, methods will be perfected for the winter stockpiling of wet ore. This would materially reduce the investment in concentration plants per ton of yearly capacity, an item that will become more important when more elaborate plants are constructed for the concentration of the more complex ores.

Conclusions

Great changes undoubtedly will occur in the Lake Superior iron-ore districts during the next 25 years. The engineers will be called upon to further improve existing operations and perfect new methods for mining, transporting, and beneficiating iron ore and converting this material into usable forms. The quantity of iron ore that can be secured from the Lake Superior district is almost inexhaustible and there is no reason why it should be conserved. It seems to be the opinion now that we have more iron ore and smelting capacity than will ever be needed, but new developments undoubtedly will be made, such as the use of cast iron in the paving industry as previously mentioned, which eventually will stimulate the demand for iron ore. This will increase the demand for beneficiated ore and greatly stimulate an industry that now seems to be lagging. At the present time, the Lake Superior district is equipped to produce annually several times the tonnage of ore required, but the work of preparing for the time when the low-grade ores must furnish the bulk of material for the steel industry goes steadily forward. Political as well as engineering difficulties must be overcome, but progress is being made perhaps more in the direction of science than in politics. Both are receiving considerable attention, however, with the result that there need be no grave concern about a shortage of iron ore from the Lake Superior district.

Concentration of Tungsten Ore by the Nevada-Massachusetts Company

By Ott F. Heizer,* Member A.I.M.E.

The mines and the mill of the Nevada-Massachusetts Co. are on the east slope of the Eugene Mountains, in Pershing County, Nevada, 8 miles northwest of Mill City, a station on the Southern Pacific R.R. and on the Victory Highway. Fig. 1. Supplies and concentrate are transported by truck over an automobile road of easy grade. The property is at an average elevation of 5500 ft. and climatic conditions are generally favorable for continuous operation throughout the year.

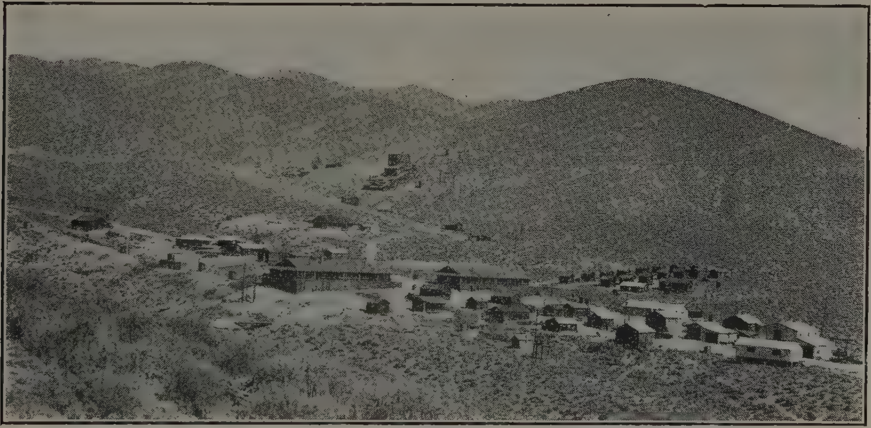

Fig. 1.—Nevada-Massachusetts Company mining camp.

The ore treated is supplied by two adjoining mines, the Stank and the Humboldt. Physical and mineral characteristics of the ores produced by these two mines are substantially similar, although variations occur in hardness and mineral distribution.[1]

The economic mineral is scheelite ($CaWO_4$), composed, when pure, of 80.6 per cent tungstic trioxide (WO_3) and 19.4 per cent lime (CaO).

Manuscript received at the office of the Institute Sept. 8, 1934.

* General Manager, Nevada-Massachusetts Co., Mill City, Nevada.

[1] F. L. Hess and E. S. Larsen: Contact-metamorphic Tungsten Deposits, U. S. Geol. Survey *Bull.* 725 (1921).

P. F. Kerr: Tungsten Deposits Near Mill City, Nevada. Nevada Bur. Mines *Bull.* (1934) **28**, No. 2.

O. F. Heizer: U. S. Bur. Mines *Inf. Circ.* 6284 (1930).

O. F. Heizer: U. S. Bur. Mines *Inf. Circ.* 6280 (1930).

The ore is mined from highly metamorphosed thin limestone member of a siliceous sedimentary series that has been intruded by granodiorite. These veinlike measures, though considerably faulted and distorted, are traceable for about 7500 ft. on the surface and show no material change in grade or mineral composition on the downward extensions to the present level of 900 ft. The scheelite occurs in unevenly disseminated grains varying from a fraction of a millimeter to several decimeters in cross-section. Seventeen minerals have been identified in the ore, which also contains a small amount of silver and gold, which is not recovered. The following may be considered a typical analysis of the mill feed:

	Per Cent		Per Cent		Per Cent
WO_3	1.24	S	1.44	As_2O_3	Trace
SiO_2	47.06	P	0.24	SnO_2	Trace
Fe_2O_3	10.89	MgO	0.48	CO_2	5.34
Al_2O_3	8.76	CuO	0.06	ZnO	0.08
MnO	1.01	MoO_3	0.05	NiO_2	0.02
CaO	23.52	Bi_2O_3	0.03		

Specific gravity 3.1

From a milling standpoint, the minerals principally considered apart from the scheelite are quartz, garnet, epidote, zoisite, vesuvianite and pyrite, which range from 6 to 7.5 in hardness, and in grinding tend to pulverize the scheelite, which varies from 4.5 to 5 in hardness, is quite friable and slimes readily. In panning the ore or tailing, if there is a question as to the identity of a heavy white mineral, pressing the mineral against the bottom of the pan with the thumb nail is sufficient to crush scheelite, producing a momentary milky cloud in the water.

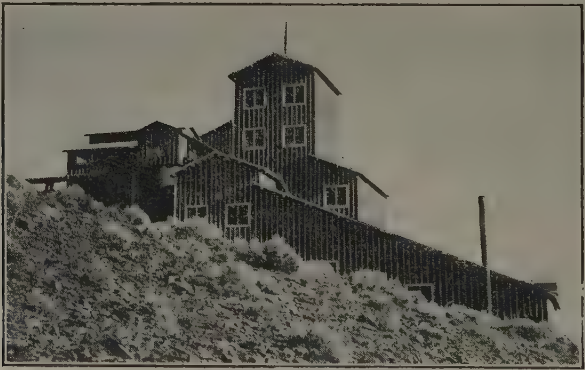

Fig. 2.—Nevada-Massachusetts mill.

Step reduction by rolls was selected as the best means of liberating the maximum quantity of scheelite with a minimum production of slimed mineral. The mill (Fig. 2) was erected during the latter part of 1918 under the stimulus of the war demand for tungsten alloys. Soon after the signing of the Armistice it was shut down because of inability to compete with cheap foreign ores and products, which were then dumped on the market.

In September, 1922, a tariff of 45¢ per pound was placed on tungsten (this tariff was increased to 50¢ per pound in 1928) and in 1924 operations were again resumed by leasers. In 1925 the Nevada-Massachusetts Co. began operating. Since that time the mill production has been as follows:

Total tons of ore milled	380,293
Scheelite concentrates produced	7,407,852 lb. or 3,703.926 tons
Tungstic acid (WO_3)	259,348.9 units
Average pounds recovery WO_3 per ton treated	13.64
Average grade of concentrate produced, per cent WO_3	70.02
Mill production, Jan. 1 to Aug. 1, 1934:	
Total tons milled	40,399
Scheelite concentrate produced	892,665 lb. or 446.33 tons
Tungstic acid (WO_3) produced	655,662 lb.
WO_3 in units of 20 lb	32,783 units
Average tons milled daily	192.3
Average recovery WO_3 per ton	16.23 lb.
Average grade of concentrate produced, per cent WO_3	73.45

All concentrate sold is guaranteed to comply with the following specifications as to minimum WO_3 content and maximum impurities:

	PER CENT		PER CENT		PER CENT
Tungstic trioxide (WO_3)	65–70	Sulfur	0.75	Antimony	Trace
Tin	Trace	Phosphorus	0.05	Bismuth	Trace
Copper	0.05	Arsenic	Trace		

The following concentrate analysis may be considered as representative of the present mill product:

	PER CENT		PER CENT		PER CENT
WO_3	73.60	S	0.356	Fe_2O_3	1.28
Cu	Trace	Sb	Trace	MnO	0.32
Ni	Trace	P	0.018	Al_2O_3	1.16
As	0.013	CaO	20.62	Mo	0.28
Sn	Trace	SiO_2	2.30		

Although the capacity of the mill has been doubled, the only material changes in the flow sheet were those made necessary when mining operations entered the sulfide zone and consist mainly of flotation treatment of the concentrate for the elimination of fine pyrite and nonmagnetic metal sulfides and the roasting and magnetic separation of the coarser metal sulfides.

FLOW SHEET

The ore is delivered from the mines over a surface track by a 7-ton Plymouth gasoline locomotive drawing six cars holding 5 tons each. The track gage is 24 in. and there is a 4 per cent grade in favor of the load. All tramming is done during the day shift only. Two parallel bins of 250 tons each receive the ore. (Fig. 3.) Eight R. & P. gates, 24 by 30 in., control the discharge from the two bins on to a variable-speed, clutch-controlled, steel apron conveyor between the two bins, which discharges to a grizzly. A Blake-type crusher reduces the oversize, which joins the grizzly undersize on a belt conveyor discharging into a 14-in. by 48-ft. belt and bucket elevator equipped with 12 by 6-in. buckets spaced 16 in. apart. The conveyor speed is 280 ft. per minute and the elevator speed is 300 ft. per minute. This elevator discharges into a revolving trommel, 3⁄16-in. steel plate, 9⁄16-in. holes, 20° slope and 19 r.p.m. The trommel oversize drops into a set of Garfield rolls, set to ½ in., 63 r.p.m., operating in closed circuit with the conveyor, elevator and trommel.

A set of rolled chrome-steel shells on this work reduces 56,000 tons of ore, showing a steel consumption of 0.034 lb. per ton.

The trommel undersize passes to a belt conveyor equipped with a hand-propelled tripper, which discharges into two cylindrical redwood-stave tank bins.

A right-hand mechanical feeder attached to the bottom of each of these tanks feeds on to a conveyor, speed 300 ft. per minute, which delivers the ore to two 14-in. by 55-ft. belt and bucket wet elevators, speed 300 ft. per minute, which discharge into one of two trommels equipped with No. 23 Ton-Cap screen, ⅛ by ¼-in. openings. The percentage of openings is 38.

The trommel oversize passes to a set of rolls, 95 r.p.m., operating in closed circuit with the wet elevators and trommel. The set of these rolls is varied with the amount of the circulating load. If the load is seen to be building up, the rolls are set up a little closer. The undersize passes to a duplex Callow screen equipped with 14-mesh phosphor-bronze Ton-Cap screen. Two sets of rolls receive the Callow-screen oversize, grinding in closed circuit with the elevators, trommel and Callow screen.

Cast-steel roll shells grinding the trommel oversize last approximately five months and show a steel consumption of 0.063 lb. per ton. Cast-steel shells on the rolls grinding the Callow-screen oversize and table middling last approximately seven months and show a steel consumption of 0.045 lb. per ton.

The 14-mesh Callow-screen undersize flows to a single-spigot hydraulic classifier, which delivers coarse sands through a spigot to six Wilfley and Deister-Overstrom tables. The overflow goes to a second single-spigot hydraulic classifier, which delivers fine sand through the spigot to five Wilfley and Deister-Overstrom tables and an overflow which goes to three 8-ft. Callow cones. The Callow-cone goosenecks discharge into a simple nonhydraulic classifier, which roughly separates the coarser and heavier from finer and lighter products. The coarser and heavier part of the slime passes through a spigot and is dressed on two Wilfley tables.

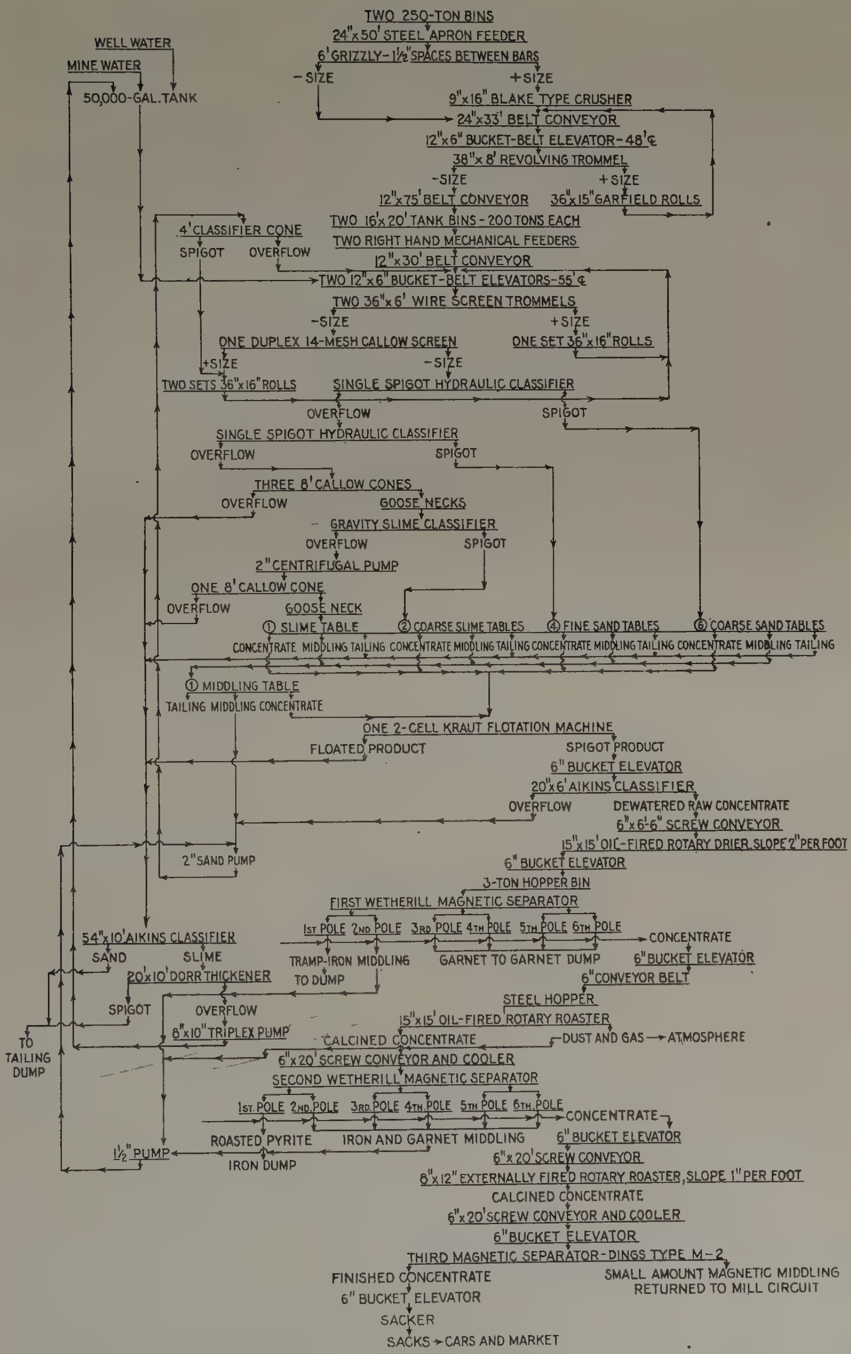
TWO 250-TON BINS
24"x50' STEEL APRON FEEDER
6' GRIZZLY-1½" SPACES BETWEEN BARS
WELL WATER
MINE WATER
50,000-GAL. TANK
- SIZE
+ SIZE
9"x16" BLAKE TYPE CRUSHER
24"x33' BELT CONVEYOR
12"x6" BUCKET-BELT ELEVATOR-48'℄
38"x 8' REVOLVING TROMMEL
-SIZE
+SIZE
12"x75' BELT CONVEYOR
36"x15" GARFIELD ROLLS
TWO 16'x20' TANK BINS - 200 TONS EACH
TWO RIGHT HAND MECHANICAL FEEDERS
4' CLASSIFIER CONE
SPIGOT
OVERFLOW
12"x30' BELT CONVEYOR
TWO 12"x6" BUCKET-BELT ELEVATORS-55'℄
TWO 36"x 6' WIRE SCREEN TROMMELS
-SIZE
+SIZE
ONE DUPLEX 14-MESH CALLOW SCREEN
ONE SET 36"x16" ROLLS
+SIZE
-SIZE
TWO SETS 36"x16" ROLLS
SINGLE SPIGOT HYDRAULIC CLASSIFIER
OVERFLOW
SPIGOT
SINGLE SPIGOT HYDRAULIC CLASSIFIER
OVERFLOW
SPIGOT
THREE 8' CALLOW CONES
OVERFLOW
GOOSE NECKS
GRAVITY SLIME CLASSIFIER
OVERFLOW
SPIGOT
2" CENTRIFUGAL PUMP
ONE 8' CALLOW CONE
OVERFLOW
GOOSE NECK
① SLIME TABLE
② COARSE SLIME TABLES
④ FINE SAND TABLES
⑥ COARSE SAND TABLES
CONCENTRATE MIDDLING TAILING CONCENTRATE MIDDLING TAILING CONCENTRATE MIDDLING TAILING CONCENTRATE MIDDLING TAILING
① MIDDLING TABLE
TAILING MIDDLING CONCENTRATE
ONE 2-CELL KRAUT FLOTATION MACHINE
FLOATED PRODUCT
SPIGOT PRODUCT
6" BUCKET ELEVATOR
20'x6' AIKINS CLASSIFIER
OVERFLOW
DEWATERED RAW CONCENTRATE
6"x 6'-6" SCREW CONVEYOR
15"x15' OIL-FIRED ROTARY DRIER, SLOPE 2" PER FOOT
6" BUCKET ELEVATOR
2" SAND PUMP
3-TON HOPPER BIN
FIRST WETHERILL MAGNETIC SEPARATOR
1ST POLE 2ND POLE 3RD POLE 4TH POLE 5TH POLE 6TH POLE
CONCENTRATE
54"x10' AIKINS CLASSIFIER
SAND
SLIME
TRAMP-IRON MIDDLING
TO DUMP
GARNET TO GARNET DUMP
6" BUCKET ELEVATOR
20'x10' DORR THICKENER
6" CONVEYOR BELT
STEEL HOPPER
SPIGOT
OVERFLOW
15"x15' OIL-FIRED ROTARY ROASTER
8"x10" TRIPLEX PUMP
CALCINED CONCENTRATE
DUST AND GAS → ATMOSPHERE
TO TAILING DUMP
6"x20' SCREW CONVEYOR AND COOLER
SECOND WETHERILL MAGNETIC SEPARATOR
1ST. POLE 2ND. POLE 3RD. POLE 4TH. POLE 5TH POLE 6TH. POLE
CONCENTRATE
ROASTED PYRITE
IRON AND GARNET MIDDLING
6" BUCKET ELEVATOR
1½" PUMP
IRON DUMP
6"x20' SCREW CONVEYOR
8"x12" EXTERNALLY FIRED ROTARY ROASTER, SLOPE 1" PER FOOT
CALCINED CONCENTRATE
6"x20' SCREW CONVEYOR AND COOLER
6" BUCKET ELEVATOR
THIRD MAGNETIC SEPARATOR-DINGS TYPE M-2
FINISHED CONCENTRATE
SMALL AMOUNT MAGNETIC MIDDLING RETURNED TO MILL CIRCUIT
6" BUCKET ELEVATOR
SACKER
SACKS → CARS AND MARKET

FIG. 3.—FLOW SHEET OF TUNGSTEN CONCENTRATOR AT MILL CITY.

The finer and lighter part flows to a centrifugal pump, which delivers it to a single Callow cone. The gooseneck product from this cone is dressed on a Deister table. All Callow-cone overflows go into the tailing launder.

All tables produce a concentrate, middling and tailing. All table middlings flow to a single Deister-Overstrom table for redressing. A broad middling flows from this table to a centrifugal pump, which delivers it to a cone above the fine rolls. The spigot from this cone drops into the fine rolls grinding the Callow-screen oversize and the cone overflow joins the feed and circulating load in the wet elevators.

The raw concentrate flows to a two-cell Kraut flotation machine, which eliminates the fine sulfide minerals. The flotation tailing, consisting of the scheelite together with coarse pyrite and garnet, flows into a bucket elevator discharging into a small Akins classifier, for dewatering. The overflow from the classifier joins the table middling and goes back into the mill circuit. The raw concentrate, averaging 50 per cent scheelite, now reduced to 7 per cent water, drops into a screw conveyor, and is delivered into the upper end of an oil-fired rotary drier running at 10 r.p.m., designed and built on the property. Oil consumption is estimated at one gallon per hour. The dried concentrate drops into the boot of a belt and bucket elevator, which discharges into a hopper bin. The operation up to this point is continuous throughout the three shifts. The roasting and magnetic separation is confined to two shifts.

The dried concentrate that has accumulated in the hopper bin is fed evenly on to the belt of the first magnetic separator. Continuous current is supplied by a motor-generator set consisting of a 10-hp., 1140-r.p.m., 440-volt, alternating-current motor belted to a 6-kw., 125-volt, 48-amp., 1180-r.p.m. generator. Magnetic characteristics of the first magnetic separator are as follows:

POLES	AMPERE TURNS	AMPERES	VOLTS
2	30,000	1.4	120
2	60,000	11.4	120
2	100,000	21.4	120

Tramp iron is removed by the first pole and goes to the dump, a small quantity of pyrite granules that have been reduced in drying is removed by the second and third poles and delivered to the middling pump and returned to the mill circuit. The fourth, fifth and sixth poles remove the bulk of the garnet, which is wheeled to a separate garnet dump.

The discharged raw concentrate from the first Wetherill magnetic separator passes into a belt and bucket elevator and from there to a belt conveyor and into a steel hopper at the feed end of the rotary oil-fired roaster. This roaster consists of a tube 15 in. in diameter by 15 ft. long, lined with firebrick and tile, slopes 2 in. to the foot, rotates at 10 r.p.m. and consumes an estimated 1½ gal. of 27° Bé. oil hourly. Time of contact in the roaster is approximately five minutes. The temperature carried is sufficient to ignite the sulfur only. The aim is to produce a magnetic sulfide on the surface of the pyrite grains. If the roast is carried too far, only faintly magnetic Fe_2O_3 results. It has not proved feasible to dry and roast in one operation on account of the low fusibility of some of the garnets, which results in a tendency to clinker. Dust and gasses are collected from the roaster stack and hot calcine discharge by a 36-in. motor-driven exhauster and delivered to a 30-in. by 8-ft. cyclone dust precipitator. Collected dust, sweepings, middlings, etc., are pumped to the middling pump, which returns these products to the mill circuit.

The hot calcined concentrate is discharged into a steel screw conveyor, where it is spray-quenched with a small amount of water and cools in transit to the second Wetherrill magnetic separator. This machine is similar to No. 1 magnetic separator and is equipped with a similar direct-current motor-generator set. The magnet characteristics are as follows:

Poles	Ampere Turns	Amperes	Volts
2	30,000	3.5	120
2	60,000	11.4	120
2	100,000	21.4	120

The first pole removes the reduced iron sulfide, which is wheeled to a separate iron dump. The second, third, fourth, fifth and sixth poles remove a middling product composed of both iron and garnet, with attached scheelite, which goes back into the mill circuit. The concentrate goes into a belt and bucket elevator and from there to a steel screw conveyor, which delivers it to an externally oil-fired rotating tube, at 6 r.p.m., where it is dead-roasted to eliminate residual sulfur and carbon. Estimated oil consumption is 1½ gal. hourly. The calcined concentrate is discharged into a steel screw conveyor where it cools in transit to a belt and bucket elevator. This elevator discharges into a Dings magnetic separator, type M-2, which removes a very minute quantity of magnetic material. The finished concentrate is elevated by a belt and bucket elevator to a hopper, from which it flows through a 1¼-in. pipe into a sacker.

The finished concentrate is double sacked, canvas inside of burlap. The sacks empty are 14 by 24 in., holding, when full, approximately 0.55 cu.ft. of concentrate and weighing 120 lb. The average car shipment is 70,000 lb. of concentrate.

Mill tailings flow through a launder to an outside building. A 54-in. by 10-ft. Akins classifier divides it into sand and slime products in the ratio of 75 and 25. The slime flows into a 20 by 10-ft. Dorr thickener, which produces a clear over-flow and a spigot product averaging 3.7:1 density. This spigot product joins the classifier sands and carries the tailing down the dump with an average pulp density of 1.15:1. The Dorr overflow is pumped up to a 50,000-gal. tank above the mill, where it joins water pumped from the mines and such additional water as may be required from the valley well.

The mines supply most of the water used in the mill. Such additional water as may be required is supplied from a 56-ft. well sunk in the valley to the east of the property. This well is the source of the domestic water supply.

The water is pumped into a tank by a centrifugal pump on the well casing and is relayed to a 100,000-gal. tank on the property in two lifts by two 7 by 10-in. triplex pumps, each belt-driven by a 50-hp., 870-r.p.m., 440-volt motor. The centrifugal pump is direct connected to a 10-hp., 1700-r.p.m., 440-volt motor. The total difference in elevation is 776 ft. and the total length of the 6-in. redwood-stave pipe line is 21,150 ft. The capacity of the pumping plant is 250 gallons per minute. The cost of the well water is about 20¢ per 1000 gallons.

Power is purchased from the Sierra Pacific Power Co. The generating plant is at Lahontan Dam of the Truckee Irrigation Project, 140 miles distant. The power is transmitted at 60,000 volts and stepped down to 6,600 volts at the mine substation for distribution around the property. It is finally dropped to 440 volts for all motors.

A six-spigot Fahrenwald sizer, together with a 30-ft. Dorr thickener, is now being installed to replace present and proposed slime treatment. Experimental work in the flotation of the slimed scheelite is also being conducted.

A total of 12 men, including the superintendent and repairmen, are required to operate the mill on a three-shift basis.

Milling Practice at Buchans Mine, Buchans, Newfoundland

By G. A. Hellstrand* and P. W. George,† Member A. I. M. E.

In 1915, H. A. Guess, Vice President of American Smelting & Refining Co., in charge of its Mining Department, learned that the Anglo-Newfoundland Development Co., Ltd., a pulpwood and paper-mill enterprise, had found upon its large concession in Newfoundland, and had opened up by shaft to the third level and some drifting, a lenticular orebody indicating about 100,000 tons, as an extremely fine-grained sulfide ore, averaging about 0.05 oz. Au, 4 oz. Ag, 1.5 per cent Cu, 10 per cent Pb, 18 per cent Zn; and that having sent parcels of this ore to Minerals Separation, Ltd., and various other metallurgical testing concerns without obtaining any success in commercial separations thereof, had discontinued work thereon and let the workings fill.

Mr. Guess wrote to the general manager of that company and obtained a few hundred pounds of the typical ore, which he sent to his company's metallurgical testing laboratory at Flat River, Mo., outlining various flotation tests desired thereon, in an endeavor to obtain separation into a commercial lead concentrate and a zinc concentrate. But the art of flotation had not then progressed far enough to treat such ore, the galena and zinc blende being so fine grained that grinding to about 300 mesh was required to release the mineral particles in their gangue. Mr. Guess had flotation and other tests continued upon this ore from time to time with whatever new reagents and flotation devices were evolved as time went on, and finally, in 1925, he succeeded in effecting in his company's testing plant, then at Wallace, Idaho, separations into lead concentrates and zinc concentrates of satisfactory commercial grade.

Mr. Guess then proceeded to unwater, examine and sample the property and made with the owners a partnership operating agreement upon the mine itself and a considerable area surrounding it, and in the early summer of 1926 began to prospect for additional orebodies, utilizing for such prospecting geophysical methods under the direction of Hans Lundberg, methods which up to that time had been little used in mining in the western hemisphere. This geophysical work found two large flat-lying orebodies of a type precisely similar to the smaller body of the original discovery. The larger orebody, some 3500 ft. west of the original discovery, was named "Lucky Strike," and the other, 2000 ft. east of the original, was named "Oriental."

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* Mill Superintendent, Buchans Mining Company Limited, Buchans, Newfoundland.

† Manager, Buchans Mining Company Limited.

The aggregate of these two newly discovered orebodies, after development by diamond drilling and underground work, represented about 6,000,000 tons averaging approximately as follows: Au, 0.05 oz.; Ag, 3.5 oz.; Cu, 1.5 per cent; Pb, 8.0 per cent; Zn, 17.0 per cent; Fe, 7.8 per cent.

Copper values in each of these orebodies were to some extent segregated into definite bodies of higher grade copper, the aggregate of which in the Lucky Strike and Oriental orebodies showed some 600,000 tons averaging approximately: 0.02 oz. Au, 2.2 oz. Ag, 5.9 per cent Cu, 2.1 per cent Pb, 12.1 per cent Zn, 23.8 per cent Fe.

Diamond drilling and underground development on the Lucky Strike orebodies were done in the fall of 1926 and in 1927. In the fall of 1928 a crushing plant of 1200 tons daily capacity and a flotation plant of 500 tons capacity were completed and put in operation.

Other features of the enterprise were the construction of a 22-mile railway from the plant to the Anglo-Newfoundland Development Company's Millertown branch; a 2500-hp. hydroelectric plant; steel and concrete storage sheds for concentrates at Buchans and at the port of Botwood, equipped with cranes, and equipment of two gantry cranes at the loading pier at Botwood.

In the summer of 1930 the addition of a second unit for the Buchans mill was begun and this was completed and put in operation July 15, 1931, giving a monthly milling capacity of approximately 37,000 tons, and additional power was brought by transmission line from the Deer Lake powerhouse of International Power & Paper Co. of Newfoundland, Ltd.

Buchans mine is 6 miles northwest of the 30-mile long Red Indian Lake in the west central part of Newfoundland, and at an elevation of 900 ft. above sea level. By railway it is 274 miles from Port-aux-Basques, 347 miles from St. John's and 92 miles from the port of Botwood.

The Buchans operations are conducted by Buchans Mining Co. Ltd., a Newfoundland corporation of which H. A. Guess is president. Its partner in the enterprise is Anglo-Newfoundland Development Co., Ltd., one of Lord Rothermere's companies, of which Frank J. Humphrey is president and George F. Laycock, who was identified with the early development of the original Buchans orebody, some 20 years ago, is the mining engineer.

The Enlarged Mill

The flow sheet is presented in Fig. 3. Mill buildings are grouped on a flat site adjacent to the main shaft at Lucky Strike orebody. They are of steel and concrete construction throughout. The roofs and walls

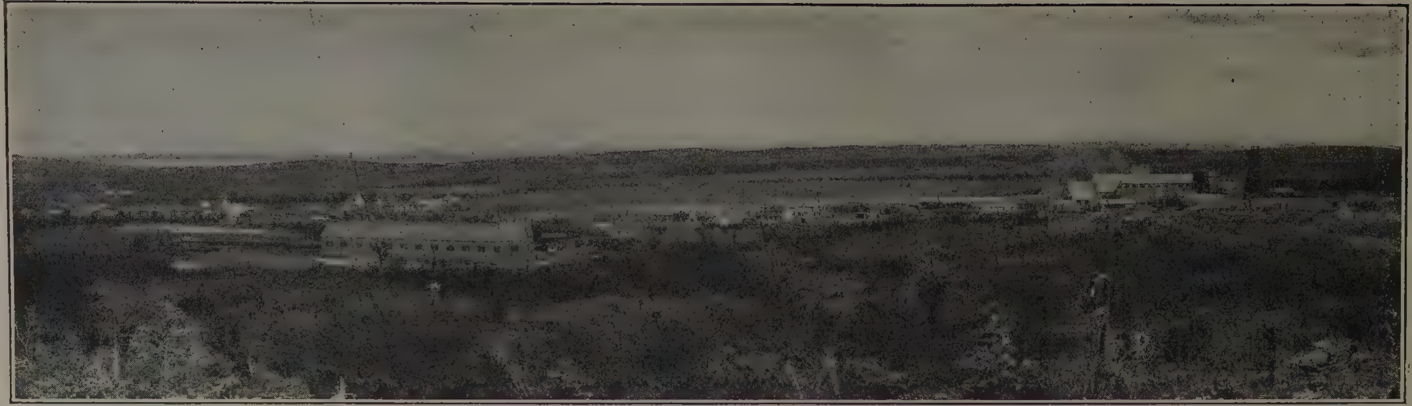

FIG. 1.—PANORAMA OF BUCHANS, NEWFOUNDLAND, FROM NORTHWEST.
Mill and Lucky Strike main shaft at right, concentrate storage sheds in foreground at left, Red Indian Lake in background at left.

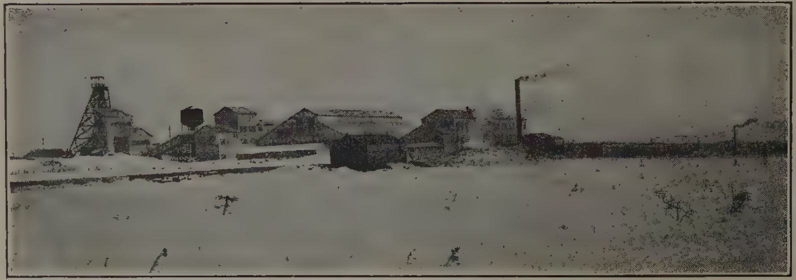

FIG. 2.—BUCHANS MILL FROM SOUTHWEST.

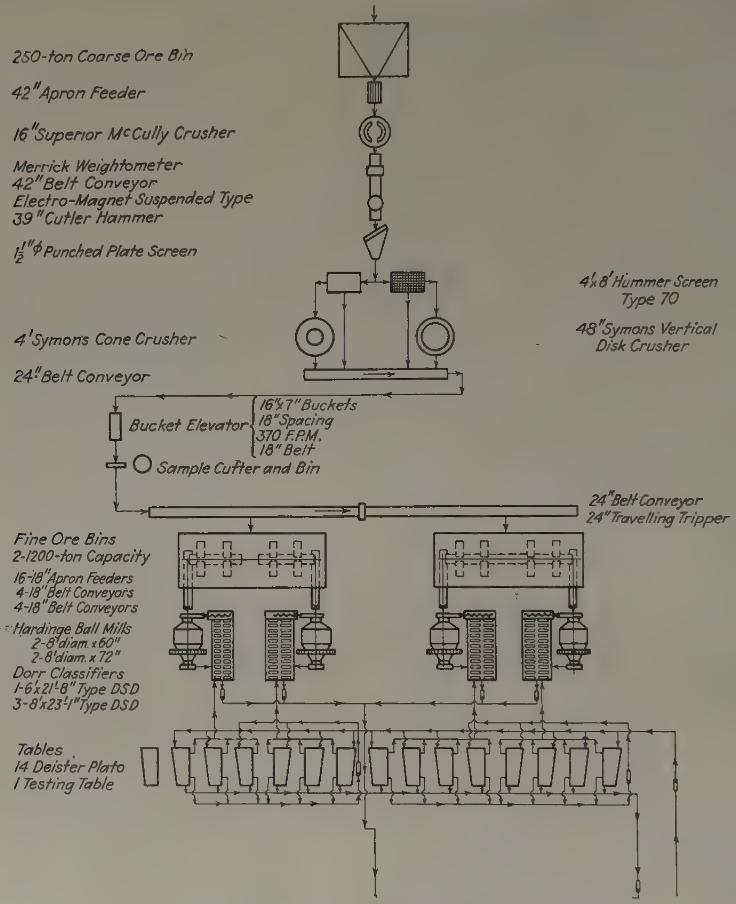

FIG. 3.—FLOW SHEET, BUCHANS MILL.

are covered with single sheets of corrugated transite, an asbestos product manufactured by Johns Manville Corporation. The coarse-ore bin and the primary crushing unit are at the shaft in a separate structure connected by enclosed conveyor runway to main building, which contains the secondary crushing units, fine-ore bins, grinding and flotation units. Dewatering, filtering and drying of concentrates are carried out in another building. The two storage sheds for concentrates are also of steel and concrete construction, with galvanized iron sheets for roofs and walls.

CRUSHING

The ore mined, after having passed through grizzlies of 11-in. opening, is hoisted and dumped into a coarse-ore bin which has a nominal capacity of 250 tons.

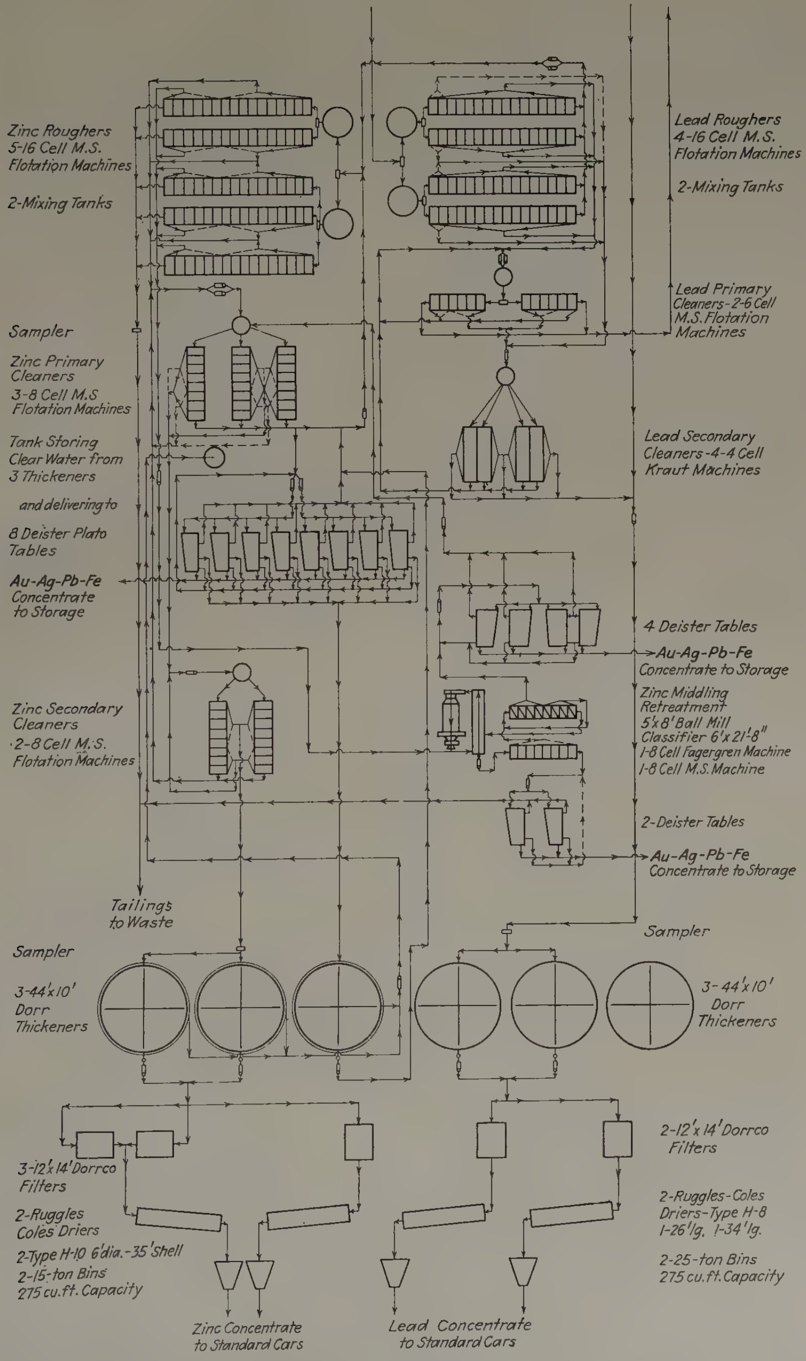

FIG. 3.—(*Continued.*)

Glory-hole system of mining accounts for practically all of the ore mined from April to December. There is more or less heavy run-off when accumulation of snow in the glory holes melts in the early spring. This is also a season of frequent heavy rains. Aside from these comparatively short periods when wet ore is experienced, crushing conditions are favorable, with just sufficient moisture to prevent excessive dusting. Crushing operations are on the basis of three shifts to meet ore-hoisting arrangement. Actual crushing time averages under 15 hr. a day. This corresponds to a crushing rate of 80 tons per hour. The maximum capacity of the crushing plant is approximately 120 tons an hour.

Crushing is divided into two stages. The ore is withdrawn from the bin by 42-in. apron feeder and fed directly into 16-in. McCully gyratory crusher over a grizzly of 3 in. opening. The grizzly undersize and the crusher product are delivered by a 42-in. belt conveyor to the junction box at the head of the secondary crushing units. The 42-in. conveyor, which has a speed of 33 ft. per minute, was originally intended to serve as an ore-sorting belt but in the current operations only wood picking is carried on at this point. The conveyor is equipped with Merrick weightometer, also 36 by 44-in. Dings magnetic head pulley and 39-in. Cutler-Hammer suspended magnet. The secondary crushing units consist of one 4-ft. Symons disk crusher and one 4-ft. Symons cone crusher arranged in parallel. The fines are removed from the feed to the disk crusher by a type 60 Hum-mer screen, 4 by 8 ft. A punched plate serves similar purpose for the cone crusher. Both the primary and secondary crushers are driven by 75-hp. motors.

The crushed products, together with fines, are delivered by a short 24-in. conveyor to an elevator, which in turn discharges on to a 24-in. conveyor. A bucket and chain sampler is installed at this point. The conveyor has a traveling tripper, for distribution of ore to two rectangular bins with double V-shaped bottom. Eight apron feeders to each bin insure maximum available capacity. The two bins are each 19 by 46½ ft. in plan and 28½ ft. from the center of the apron feeder to the tripper conveyor. They have combined capacity of 2400 tons. This capacity was obtained in part by remodeling the original fine ore bin, which had 45° bottom and a single feeder directly discharging into each of the two ball mills.

A representative screen analysis of the ore delivered to the fine-ore bin follows:

Screen size, in.	+1.050	+0.742	+0.525	+0.371	+0.263	−0.263
Cumulative per cent	1.7	9.7	27.3	49.8	62.2	37.8

GRINDING AND CLASSIFICATION

Reduction of the crusher product to flotation sizes, 90 per cent minus 200 mesh, is accomplished in one stage. A system of 18-in. conveyors delivers the ore to two 8 ft. by 60-in. and two 8 ft. by 72-in. Hardinge ball mills, the latter being the new installation. Fig. 4 shows the general arrangement. The two 8 ft. 72-in. mills and one of the 8 ft. by 60-in. mills are each in closed circuit with an 8 ft. by 23 ft. 1-in. type D Dorr classifier. Originally both 8 ft. by 60-in. mills were in closed circuit with 6 ft. by 21 ft. 8-in. classifiers, but at the time of the enlargement of the mill one of these classifiers was moved to the zinc-middling regrind circuit, which was put into operation at that time, and replaced by an 8-ft. classifier.

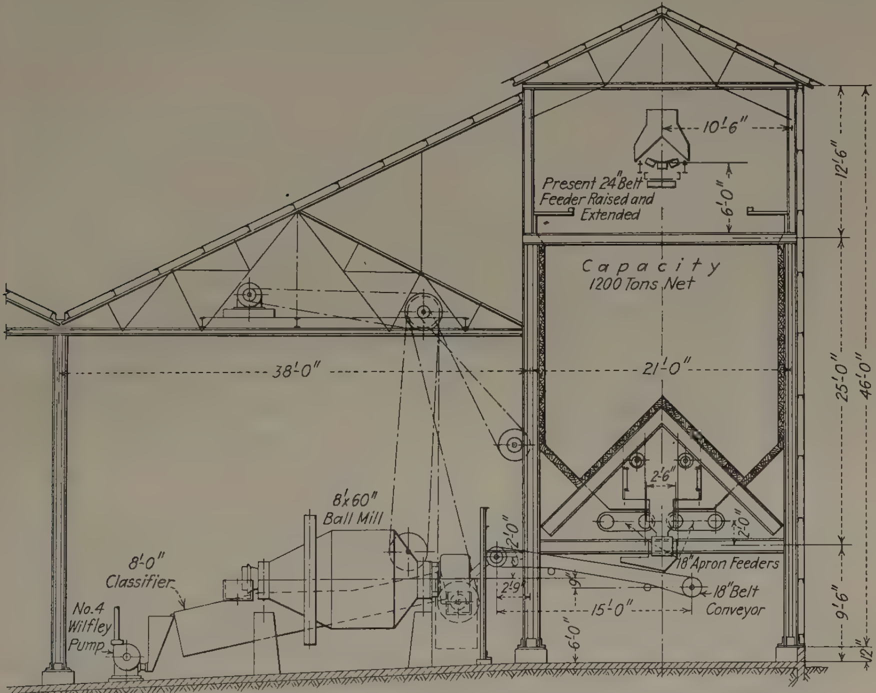

FIG. 4.—ARRANGEMENT OF FINE-ORE BIN AND GRINDING UNITS, BUCHANS MILL.

The 8 ft. by 72-in. ball mills are driven by 225-hp. synchronous, 50-cycle, 440-volt motors of 300 r.p.m. speed; the 8 ft. by 60-in. mill drives consist of 225-hp. synchronous motors of 429 r.p.m. speed. Falk couplings and Herringbone gears transmit the power and reduce the speed of the mills to 18.5 r.p.m. When grinding to 90 per cent minus 200 mesh the capacity per 24 hr. of the 8 ft. 60-in. mill is 290 tons of original feed; the capacity of the 8 ft. by 72-in. mill is 340 tons. The circulating loads average 500 to 600 per cent. The consumption of grinding balls (of forged chrome-manganese steel) averaged 1.05 lb. per ton of original feed during the last 12 months. Two-thirds of the balls used are 3½ in.; the remainder are 4 in. The liners are of manganese steel, wedge-bar type. The liners of the 8 ft. by 60-in. mills last about 21 months; those of the 8 ft. by 72-in. mills, 18 months, corresponding to a liner consumption of 0.145 lb. per ton of original feed. The following is a typical screen analysis of classifier overflow:

	Solids, Per Cent	Weight, Cumulative Per Cent				
		65 Mesh	100 Mesh	150 Mesh	200 Mesh	−200 Mesh
Classifier overflow..............	35	0.1	0.7	3.7	8.5	91.5

CHARACTERISTICS OF ORE TREATED AND DEVELOPMENT OF FLOTATION PRACTICE

The lead-zinc ore consists of a dense, fine-grained intergrowth of sphalerite, pyrite, galena, chalcopyrite, barite and quartz, with minor amounts of calcite in fractures through the ore, and of sericite derived from soft, schisted and altered quartz porphyry, mainly occurring in the hanging wall of the orebodies. The average assays of the ore milled, 1929 to 1933 inclusive, indicate the relative amounts of these minerals (Table 1).

TABLE 1.—*Average Assays of Ore Milled at Buchans Mine*

Year	Oz. per Ton		Per Cent							
	Au	Ag	Cu	Pb	Zn	Fe	$BaSO_4$	SiO_2	CaO	Al_2O_3
1929	0.06	4.32	0.79	11.37	19.26	5.9	36.1	3.0	3.4	1.1
1930	0.06	4.18	0.79	10.30	18.18	6.4	35.4	3.1	3.4	1.1
1931	0.06	3.98	0.76	9.60	17.90	6.4	35.0	4.5	3.4	1.6
1932	0.07	3.90	0.87	9.78	18.95	6.9	33.3	3.7	3.7	1.5
1933	0.06	3.45	1.21	9.38	20.81	8.3	28.4	3.5	3.6	1.3

Average specific gravity years 1932 and 1933, 4.5.

The character of the sulfide intergrowth varies considerably even within individual stopes; and variations of several per cent in zinc, lead and iron content of the mill feed are frequent. The dense, fine-grained ore of high pyrite and chalcopyrite content, mostly from stopes near the footwall of Lucky Strike, is particularly refractory. The best milling ore is the relatively coarser grained ore of low pyrite content in barite gangue, which is mined mainly in the stopes near the hanging wall.

Near the surface and in fractures through the orebody the ore is oxidized. A series of samples taken 3 ft. below the top of Lucky Strike showed that on an average 14.5 per cent of the lead in the ore was acetate soluble. Most of the samples contained oxides and sulfates of iron. The sphalerite showed signs of leaching. Some 6 ft. of the most oxidized part of the orebody at the surface is being stripped and stacked for future treatment. Despite this stripping 6 to 7 per cent of the lead in the mill feed is invariably acetate soluble, whether the ore is mined in underground stopes or in glory holes near the surface.

The chalcopyrite in the mine is mainly of two kinds. One, the dominantly prevailing, and generally classified as copper-zinc ore, is a fine-grained intergrowth of pyrite, chalcopyrite and sphalerite with relatively small amount of galena. This type of ore contains a smaller amount of gangue minerals than the average ore. The pyrite is fractured and scattered and a fine-grained chalcopyrite-sphalerite intergrowth fills the fractures in the pyrite and the spaces between the pyrite fragments. The other type of ore of high copper content contains coarser and less pyritic chalcopyrite intergrowth, the intergrowth occurring mainly in veinlets or in small patches in lead-zinc ore of average grade.

The chalcopyrite in the ore of the first type is not recovered in the lead flotation to any appreciable extent while maintaining the necessary selective condition required for lead-zinc separation. When the lead-zinc ore contains admixture of the second, coarser and cleaner type of chalcopyrite ore, the ratio of copper to lead in the lead concentrates may be sufficient to justify de-coppering of the lead concentrate.

However, the flotation problem is essentially selective separation of galena and sphalerite in the presence of pyrite and barite, the latter being the principal gangue mineral. The relatively large percentage of galena-sphalerite intergrowths that are not freed within the limits of practical grinding, the generally inert and refractory character of some of the sulfide minerals, due to surface oxidation, and the comparative ease with which calcite and altered sericitic schist and also barite float under intense flotation conditions, are some of the factors that determine the flotation practice and reagents on Buchans ore.

Fuller appreciation of these factors since the start of milling has resulted in development of such operating practice as: (1) tabling of the primary lead cleaner tailing, with its relatively high lead-zinc ratio, for the recovery of the more refractory galena, particularly the coarser sizes, which float in the roughing operation but will not carry through two stages of closed-circuit cleaning operation necessary to attain high-grade concentrate; (2) the introduction of regrind and separate treatment of zinc rougher middling in addition to a finer initial grind; (3) reagents highly selective with respect to gangue minerals in conjunction with cyanide, which is essential to best results in lead flotation; (4) double cleaning of both lead and zinc rougher concentrates and substantial increase in the roughing capacity. In the current operation, which is at the rate of approximately 1250 tons a day, 204 standard 24-in. Minerals

Separation Sub-A cells and 16 Kraut cells are used for lead and zinc flotation, as against a total of 64 Minerals Separation cells originally put into operation.

LEAD CIRCUIT

The overflow from each of the four classifiers is delivered by a 4-in. Wilfley pump to a common distributor, which divides the pulp between two surge tanks, 12-ft. dia. by 6 ft., ahead of the lead roughers. There are four 16-cell, 24-in. M.S. machines for roughing operation in lead flotation. Concentrate is made from all 16 cells. There is no direct return of product from any of the cells to the head of the machine. The tailing is the feed to the zinc circuit.

Two 6-cell, 24-in. M.S. machines are used for the first cleaning of the rougher concentrate and four 4-cell Kraut machines for second cleaning. Usually the first three cells of 6-cell M.S. machines make concentrate and remaining cells middling, which is returned to the machines. Concentrate from the second cleaning operation is a finished product and the tailing is returned to the head of two 6-cell M.S. machines. The primary cleaner tailing (i.e., the tailing from two 6-cell machines), instead of returning direct to the head of four 16-cell rougher machines, is pumped to Deister table circuit which consists of 10 roughing tables and four cleaning tables, the feed to the latter being the middling from roughing tables. The table tailing is returned to the classifiers, the overflow of which is the feed to the lead circuit. The concentrate is a finished product and pumped to join the flotation concentrate. The combined concentrate passes by gravity to the dewatering and filtering plant.

The benefits derived from the interposition of table concentration between the primary cleaning and roughing units vary with the predominant type of ore in the mill feed. In the flotation of Lucky Strike ores, particularly the extremely fine-grained pyritic ore from the southeast section and near the surface, the galena is decidedly inert and sluggish, and without table concentration on the primary cleaner tailing a much more intense flotation condition is necessary in the rougher circuit, and even under this condition it is not possible to attain the same over-all recovery. In fact, the grade of the final concentrate and the recovery will both be adversely affected. Table concentrates constitute approximately 15 to 25 per cent of the total lead production, depending on the character and grade of mill feed. When the galena in the ore has more or less normal floatability the net gain by table concentration at this point is relatively small. An incidental point in connection with tabling of primary lead cleaner tailing is that the ore contains some free gold and tellurides. The gold and silver content of the table concentrate is appreciably higher than that of the flotation concentrate of approximately the same lead content.

COPPER CIRCUIT

When the lead concentrate is high in copper it is pumped to a 12 by 6-ft. tank for conditioning with the required amount of sodium dichromate and soda ash—approximately 1.2 lb. of dichromate and 0.15 lb. of soda ash per ton of concentrate treated. From the tank it is distributed, without further addition of any reagent, to two roughers consisting of 4-cell Kraut machines. For primary and secondary cleaning two 10-ft. Southwestern machines are used (Fig. 5). The result of de-coppering lead concentrate is indicated in Table 2.

ZINC CIRCUIT

Following two conditioning tanks, each 12-ft. dia. by 6 ft., to which the tailing of the lead circuit is delivered by a 6-in. Wilfley pump through a central distributor, there are five 16-cell 24-in. M.S. machines, arranged in parallel, for roughing operation. The first eight cells of each rougher machine produce rougher concentrate, and the last eight cells, middling. The rougher concentrate is cleaned in two stages. The first and second cleaner units consist of three 8-cell, 24-in. and two 8-cell, 24-in. M.S. machines, respectively. The concentrate from the second cleaning operation is a finished product and is pumped to the dewatering and filtering plant.

In connection with first cleaning of rougher concentrate, a portion of the primary cleaner tailing that is returned to the rougher machines is bypassed and pumped to a table circuit consisting of eight Deister tables. A small amount of lead-iron concentrate, of rather low grade but carrying sufficient gold and silver values to justify shipment under special smelter contract, is produced at this point, the 1933 production being 329 tons assaying 0.74 oz. gold, 8.4 oz. silver, 36.7 per cent lead, 9.8 per cent zinc and 18.3 per cent iron. The table tailing is dewatered in a Dorr thickener and returned to the distributor at the head of the rougher machines.

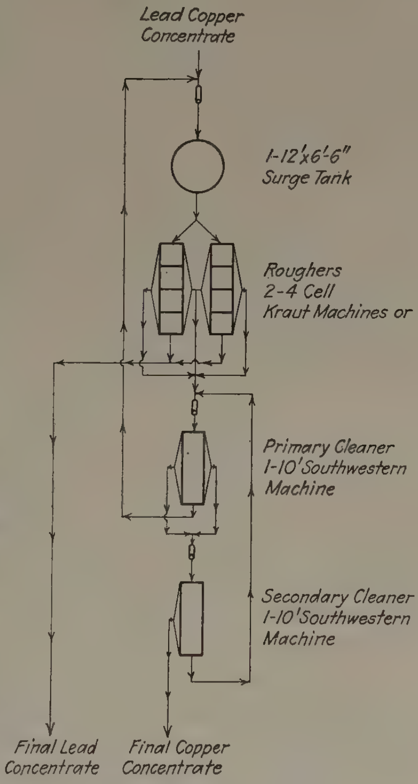
Lead Copper Concentrate
1-12'x6'-6" Surge Tank
Roughers 2-4 Cell Kraut Machines or
Primary Cleaner 1-10' Southwestern Machine
Secondary Cleaner 1-10' Southwestern Machine
Final Lead Concentrate
Final Copper Concentrate

FIG. 5.—FLOW SHEET OF COPPER CIRCUIT, BUCHANS MILL.

TABLE 2.—*Results of De-coppering Lead Concentrates One Month of* 1933

	Weight, Per Cent	Assay, Per Cent					
		Au	Ag	Cu	Pb	Zn	Fe
Bulk lead-copper concentrate	100.00	0.098	10.49	4.16	55.47	9.04	8.03
Copper concentrate	11.23	0.080	8.80	25.01	9.75	9.14	22.90
Final lead concentrate	88.77	0.10	10.70	1.52	61.27	9.02	6.15
		Recovery, Per Cent					
Bulk lead-copper concentrate		100.00	100.00	100.00	100.00	100.00	
Copper concentrate		9.19	9.43	67.55	1.97	11.36	
Final lead concentrate		90.81	90.57	32.45	98.03	88.64	

ZINC-MIDDLING REGRIND CIRCUIT

The rougher middling from the last eight cells of each rougher machine is the feed to the regrinding unit, which consists of 5 by 8 Allis Chalmers ball mill direct connected to 100-hp. motor and 6 ft. by 21 ft. 8-in. type D Dorr classifier in closed circuit. The classifier overflow is pumped to an 8-cell, 24-in. M.S. machine, which makes concentrate and tailing. The concentrate is retreated in an 8-cell Fagergren machine and after passing over four Deister tables joins the rougher concentrate from the first eight cells of the five 16-cell M.S. machines and is pumped to the primary cleaner circuit. The four tables treating Fagergren machine concentrate produce 159 tons of low-grade lead concentrate, averaging 1.69 oz. gold, 12.2 oz. silver, 23.3 per cent lead, 6.1 per cent zinc and 26.9 per cent iron in one year. The tailing of the 8-cell M.S. machine goes to waste, first passing over two Deister tables. The iron concentrate made at this point carries the highest gold value of any table concentrate made in regular operation. Production is 176 tons, assaying 3.22 oz. gold, 24.9 oz. silver, 6.8 per cent lead, 2.8 per cent zinc and 37.1 per cent iron in one year. Corduroy has been tried on this table tailing, and results indicate some possibilities of further recovery of gold, which occurs mainly in the form of tellurides.

DEWATERING, FILTERING, AND DRYING CONCENTRATES

The filter plant, aside from the portion that houses the concentrate thickeners, contains three floors. In 1930, in connection with enlargement of the mill, full advantage was taken of the height of the building to install gravity flow of the concentrate from the filters through the driers and storage bins to railroad cars. On the uppermost floor are

TABLE 3.—*Production and Metallurgical Data, Buchans Mine*

PRODUCTION

Year	Dry Tons Ore Milled	Concentrates, Tons				Ratio of Concentration	
		Lead	Zinc	Copper	Lead-iron	Lead	Zinc
1929	163,114	22,717	52,204			7.2	3.1
1930	188,299	25,150	52,815			7.5	3.6
1931	311,299	38,833	87,526			8.0	3.6
1932	438,255	53,398	131,574		342	8.2	3.3
1933	439,450	50,025	153,275	840	714	8.8	2.9

ASSAY OF PRODUCTS

Year	Lead Concentrate						Zinc Concentrate					
	Au, Oz.	Ag, Oz.	Cu, Per Cent	Pb, Per Cent	Zn, Per Cent	Fe, Per Cent	Au, Oz.	Ag, Oz.	Cu, Per Cent	Pb, Per Cent	Zn, Per Cent	Fe, Per Cent
1929	0.17	13.2	1.6	57.8	13.7	3.4	0.06	5.3	1.3	7.5	48.2	5.0
1930	0.17	14.1	1.6	59.8	12.0	4.2	0.06	4.6	1.3	5.4	50.4	4.8
1931	0.17	14.6	1.2	61.4	11.0	4.0	0.05	4.6	1.3	4.9	51.6	4.3
1932	0.19	14.1	1.3	62.0	10.7	4.2	0.05	4.4	1.3	5.3	51.6	4.2
1933	0.14	12.7	1.5	61.9	10.1	4.8	0.05	3.7	1.7	5.0	51.0	5.7
	Copper Concentrate						Lead-iron Concentrate					
1932							3.23	21.4	0.6	8.3	5.8	31.1
1933	0.06	8.9	24.9	11.1	8.1	22.8	1.58	13.4	0.7	25.6	7.0	26.9

RECOVERIES, PER CENT

Year	Lead Concentrate					Zinc Concentrate				
	Au	Ag	Cu	Pb	Zn	Au	Ag	Cu	Pb	Zn
1929	37.8	42.6	27.6	70.8	9.9	32.0	39.4	51.7	21.2	80.1
1930	35.2	44.9	26.4	77.5	8.8	27.1	30.6	44.5	14.7	77.8
1931	35.6	45.7	20.2	79.8	7.7	22.7	32.4	48.3	14.3	81.1
1932	35.8	44.1	17.5	77.2	6.9	23.7	34.0	45.3	16.3	81.7
1933	28.7	41.9	14.1	75.1	5.5	29.4	37.1	47.8	18.6	85.4
	Copper Concentrate					Lead-iron Concentrate				
1932						3.9	0.4	0.1	0.1	
1933	0.2	0.5	3.9	0.2	0.1	4.6	0.6	0.1	0.4	0.1

(Table continued on next page)

TABLE 3.—(*Continued*)

REPRESENTATIVE REAGENT COMBINATION FOR 1933

Lead Circuit		Zinc Circuit	
Kind	Lb. per Ton Ore	Kind	Lb. per Ton Ore
Cyanide	0.30	Copper sulfate	1.40
Zinc sulfate	0.40	Lime	0.10
Sodium carbonate	0.10	Cyanide[a]	0.01
Minerec A	0.10	Sodium Aerofloat	0.35
Butyl xanthate	0.13	Potassium xanthate	0.15
Dupont 24	0.015	Dupont 24	0.025

[a] Regrind unit.

installed the filters, and on the floor below it driers and concentrate storage bins, equipped with air-operated gates, controlled from the ground, for dumping the concentrates into railroad cars. Double lines of tracks are laid through the entire length of the building, one under the lead concentrate bins and another under the zinc concentrate bins, with sufficient clearance to permit the passage of a locomotive. The general arrangement of the building and the equipment is given in Fig. 6.

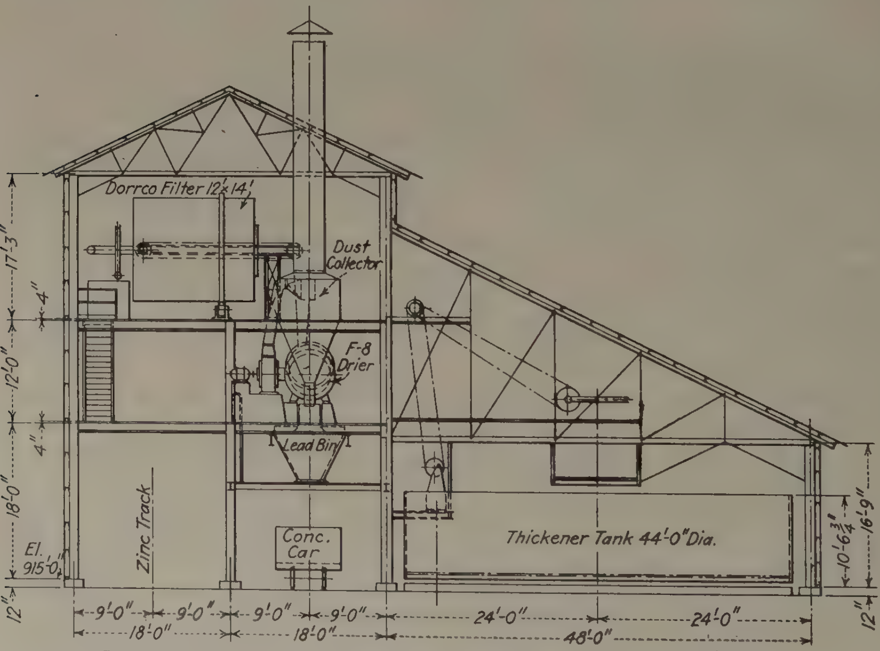

FIG. 6.—LAYOUT OF FILTER AND DRIER PLANT, BUCHANS MILL.

For dewatering of concentrates there are six 44 by 10-ft. Dorr thickeners, of which two are used for lead concentrate, one for copper concentrate, two for zinc concentrate and one for zinc cleaner tailing in connection with tabling of that product. Lime is used in settling lead and zinc concentrates and it is added at the distributor ahead of the tanks. The lead concentrate is extremely fine, averaging 99 per cent minus 200 mesh and 94 per cent minus 325 mesh; the zinc concentrate averages 89 per cent minus 200 mesh and 70 per cent minus 325 mesh. The 1933 average assays for lead and zinc concentrates follow:

Concentrate	Per Cent							
	Cu	Pb	Zn	Fe	$BaSO_4$	SiO_2	CaO	Al_2O_3
Lead	1.5	61.9	10.1	4.8	0.6	0.3	0.5	0.3
Zinc	1.7	5.0	51.0	5.7	3.2	0.7	0.7	0.5

The same general procedure is followed in filtering and drying lead and zinc concentrates. The thickened concentrate is delivered by a diaphragm pump to the feed box of a 2-in. Wilfley pump, which elevates it to a 12 by 14-ft. Dorrco filter, the center line of which is 37 ft. above the pump intake. The filter cake is discharged through a chute into a drier on the floor below. The drier discharges its product into a bin of 275-cu. ft. content, which gives available capacity of approximately 23 tons on lead concentrate and 16 tons on zinc concentrate.

A 5 by 34-ft. type H-8 Ruggles-Coles drier handles the lead concentrate. It is equipped with 7-ft. dia. cyclone dust collector and No. 21 Clarage fan, direct connected to a 750-r.p.m. 5-hp. variable-speed motor, having a 50 per cent speed variation. A 20-hp. motor drives the shell at 7 r.p.m. through a speed reducer. A duplicate unit, with a 5 by 26-ft. drier, is installed for copper concentrate and as a spare for the lead unit.

There are two filter-drier units for the zinc concentrate. The general arrangement in both units is similar to that for lead concentrate, except that in one of the units arrangements permit two filters to discharge direct into a single drier. The three Dorrco filters (Fig. 7) are of the same size, 12 by 14 ft., of standard design. At the time of installation, in 1928, three filters, now used for zinc concentrate, were equipped with screw conveyors for the discharge of the filter cake but these were replaced by belt conveyors shortly after operation began. Also, light wire backing screens were replaced by heavy punched plates. The two Ruggles-Coles driers are type H-10, 6 by 35 ft., drum speed, 7½ r.p.m., 25-hp. motor (Fig. 8). They have 8-ft. cyclone dust collectors and No. 21 Clarage fans directly connected to 750-r.p.m., 7½-hp. variable-speed motors, having a 50 per cent speed variation.

The vacuum is maintained at 23 in. of mercury either by an Ingersoll-Rand duplex vacuum pump, 27-in. dia. by 14-in. stroke, driven by 125-hp. motor or by an Ingersoll-Rand duplex pump, 20-in. dia. by 12-in. stroke, driven by 60-hp. motor. The low-pressure air for discharging the filter cake is obtained by reduction of air furnished by the main mine compressor.

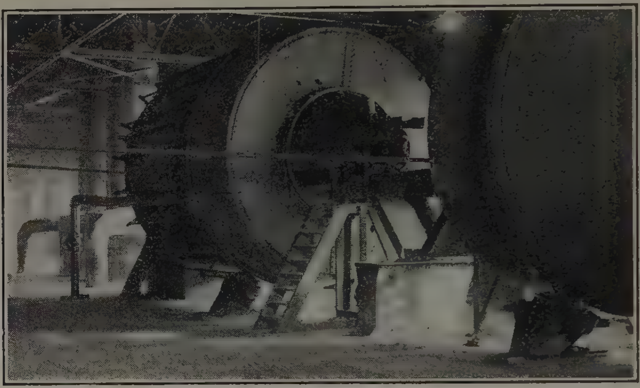

Fig. 7.—Dorrco filters.

The maximum monthly production of lead concentrates from milling operations to date is 4816 tons, or 155 tons per day. As the operating time of both the filter and drier was 30.8 days of possible 31 days, tons per filter-day and also per drier-day are approximately the same as the daily average production. During this period, moisture of the filter cake averaged 9.4 per cent and the drier discharge 4.0 per cent. The actual

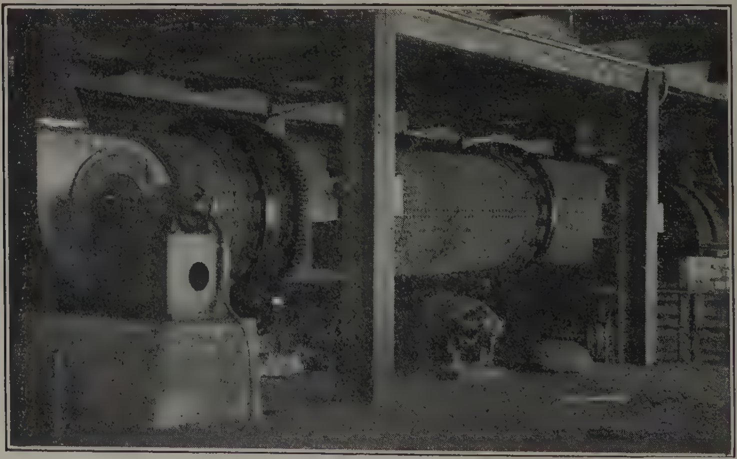

Fig. 8.—Ruggles-Coles drier.

capacity of the filter-drier unit is considerably higher than indicated by these tonnage figures.

May and June, 1934, two months of highest zinc concentrate production to date, show an average of 534 tons a day. Based on the number of filters and driers in operation during this period, the output corresponds to 295 tons per filter-day and 395 tons per drier-day. The moisture in the filtered and dried products averaged 8.9 and 5 per cent, respectively. At other periods as much as 500 tons per day has been put through the zinc drier, with slightly higher moisture in the discharge, 5.3 per cent. The tonnages for the zinc unit more or less closely approximate the actual capacity of the 12 by 14-ft. Dorrco filter and 6 by 35-ft. Ruggles-Coles drier on zinc concentrate.

Handling of Concentrate

Aside from railroad, steamship and various handling charges that are sufficiently high to justify drying, it is necessary in winter to hold the moisture low enough to prevent freezing of concentrate, which will result in added cost and delay in unloading at Botwood.

The concentrates are shipped in 25-ton, 42-in. gage, gondola railroad cars, which are covered with canvas during transportation. The port of Botwood, on the north coast of Newfoundland, 92 miles from Buchans, is generally open from the middle of May to the first week of December. During the closed season all lead concentrates and part of the zinc concentrates are stored at Botwood in a shed of steel and concrete construction, holding 40,000 tons. The shed has an overhead traveling crane and 1-cu. yd. clamshell bucket for unloading and loading cars. The bulk of the zinc concentrate is stored at Buchans in sheds. The unloading and loading of concentrates is done by crawler crane with 1½-cu. yd. clamshell bucket. A dragline scraper is used for stacking the concentrates to greater height in the sheds. The loading of concentrates from cars to ships is being done by two gantry electric cranes equipped with 1-cu. yd. clamshell buckets. The average speed of loading vessels is some 2900 metric tons per day.

Tailing Disposal

The tailing to waste is carried to Buchans River, some 4200 ft., through a covered flume, 11 by 11 in., constructed of 2-in. planks, the bottom and sides of which are lined with 1-in. boards. The grade of the flume is 2.5 per cent. Part of it is above ground, other parts are as deep as 15 ft. below the surface. Manholes are provided at distances of 250 ft. where the flume is deeply buried.

MILL ORGANIZATION

Operations are in charge of the mill superintendent, and the mill crew consists of:

Position	Number	Rate per Day
Foreman (day shift)	1	Monthly basis
Shift bosses (afternoon and night shifts)	2	$7.00
Crushing	9	$3.75 and $3.25
Ball mills	3	$3.75
Flotation	6	$4.25 and $3.75
Reagents	1	$3.75
Pumps	3	$3.50
Filtering	3	$3.75
Drying concentrates	6	$3.50 and $3.25
Loading concentrates	2	$3.25 and $3.00
Repairmen	2	$3.75
Clean-up men	3	$3.25
Electricians	1	$6.50
Samplers	3	$3.25

The assaying staff consists of three chemists and a helper and the testing staff of one engineer and a helper. Approximately 75 per cent of the total assays is represented by routine mill work and controls on concentrate shipments, the remainder by mine work.

All mechanical work, except minor routine repairs, is attended to by the mechanical department.

MILL WATER SUPPLY

Water is pumped 4400 ft. from Buchans River to a 100,000-gal. tank. The pumping equipment consists of three Allis Chalmers 6 by 5-in., type H.S. centrifugal pumps, 1450-r.p.m., 75-hp. motors, rated at 550 gal. per minute for 220-ft. head. One of the pumps is automatically controlled by float switch at the tank. For emergency use there is a fourth pump of the same capacity, driven by a gasoline engine. The water is delivered to the tank through a 10-in. pipe line buried 7 ft. below the surface. To prevent ice from forming in the tank during cold weather, eight 1½-kw. bayonet-type immersion heaters are used.

HEATING

The outside surface of the buildings, which are covered with single sheets of corrugated transite, totals 203,400 sq. ft.; the content is 3,020,000 cu. ft. The heating arrangements consist of five 170-C Heggie-Simplex boilers with grate area of 33.9 sq. ft., installed in a separate building and furnishing steam of 1 to 10 lb. pressure to radiators and unit heaters in the mill. The radiating surface required to heat the buildings in cold weather is 37,500 sq. ft. Two to three boilers are generally operated from early in December to the middle of April, with one fireman and one coal trimmer per shift. The coal consumption per year averages 1150 short tons. The annual cost of heating averages 3¢ per ton milled; the cost during the coldest months is 9¢ per ton. The number of square feet of radiation required to heat the buildings is considerably less than was estimated when the mill was enlarged in 1930–1931, owing to the heat generated by the concentrate driers and electric motors. The monthly average minimum and maximum temperatures at Buchans in years 1929 to 1934 were as given in Table 4.

TABLE 4.—*Temperatures at Buchans*

Month	1929		1930		1931		1932		1933		1934	
	Avg. Max.	Avg. Min.	Avg. Max.	Avg. Min.	Avg. Max.	Avg. Min.	Avg. Max.	Avg. Min.	Avg. Max.	Avg. Min.	Avg. Max.	Avg. Min.
Jan.				0.3	28.5	15.2	24.2	10.4	29.2	10.0	17.2	−1.7
Feb.				−2.9[a]	31.2	15.6	20.6	2.1	30.1	15.6	20.9	1.0
March			45.5[b]	20.6[b]	38.8	22.9	33.0	21.5	28.7	15.8	27.0	6.7
April			49.2	25.6	46.2	30.6	44.9	30.9	41.8	28.9	44.3	28.7
May			58.7	33.3	57.5	37.5	57.2	35.7	49.2	32.9	59.4	34.3
June			78.7	49.4	67.3	45.0	69.1	44.4	62.1	45.2	62.5	39.2
July	79.6	48.2	77.0	55.4	80.5	58.1	74.2	53.7	72.1	50.4		
Aug.	77.8	47.4	72.5	49.8	74.1	51.4	77.3	53.7	68.7	50.9		
Sept.		42.6	66.3	45.8	61.3	44.9	65.4	49.5	59.2	45.2		
Oct.		35.2	49.7	37.4	50.0	36.9	59.9	40.0	50.0	37.4		
Nov.		21.0	41.0	31.4	41.0	28.8	38.1	25.1	31.5	20.8		
Dec.		4.8	31.0	20.7	27.6	18.3	29.8	13.9	28.8	6.3		

[a] February 1–15 only. March 11–31 only.

POWER

Power is transmitted to Buchans in approximately equal amounts from two sources: from Buchans hydroelectric plant, 7800 ft. from the mill, and from the Deer Lake powerhouse of the International Power

TABLE 5.—*Consumption and Cost of Power at Buchans Mill*

	Kilowatt-hours per Month	Kilowatt-hours per Ton Milled	Cost per Ton Milled, Cents
Crushing and conveying ore to fine-ore bins	67,000	1.731	0.485
Grinding and classification	514,680	13.299	3.724
Flotation machines	520,670	13.454	3.767
Pumping (exclusive of mill water supply)	290,010	7.494	2.098
Dewatering, filtering and drying of concentrates	133,100	3.439	0.963
Lighting and miscellaneous	39,210	1.014	0.284
Total	1,564,670	40.431	11.321

and Paper Co. of Newfoundland, 45 miles distant. The power from Buchans 2500-hp. hydroelectric plant is transmitted at 6600 volts; the power from Deer Lake, approximately 2300 hp., at 66,000 volts. The current from both sources is three-phase, 50-cycle. It is transformed to 440, 220 and 110 volts for the various requirements of the mill. The substation contains 19 feeder panels for distribution of power and one panel for switching the load from one source to the other in case of interruption of delivery from either power plant. Important feeders are equipped with double-throw switches to change from one power source to the other. In 1932 and 1933 Buchans power plant averaged a running time of 99.78 per cent; the average operating time for the power transmitted from Deer Lake was 99.52 per cent. Table 5 shows the monthly consumption and cost of power for the mill during a representative month.

Cost of Milling

The average cost per ton of ore milled in 1933 is given in Table 6. The average unit costs of mill supplies are approximately 50 to 60 per cent higher than in the United States.

TABLE 6.—*Average Cost per Ton Ore Milled at Buchans in* 1933

	Labor	Supplies	Labor and Supplies
Supervision	$0.020	$	$0.020
Crushing and conveying	0.031	0.009	0.040
Grinding and classifying	0.019	0.054	0.073
Flotation reagents	0.004	0.485	0.489
All other flotation costs	0.055	0.040	0.095
Dewatering and filtering concentrates	0.011	0.018	0.029
Drying concentrates	0.019	0.040	0.059
Loading, trimming and weighing concentrates	0.010		0.010
Heating			0.028
Power			0.119
Water			0.021
Lighting			0.003
Lubricating oils and miscellaneous supplies			0.010
Testing, sampling and assaying			0.038
Total			$1.034

Milling Practice in the Tri-State Zinc-lead Mining District (Oklahoma-Kansas and Missouri)*

By S. J. Burris, Jr.,† Member A.I.M.E., C. O. Anderson,‡ Robert E. Illidge,§ Warren Howes,‖ and M. D. Harbaugh,¶ Member A.I.M.E.

Introduction

The Tri-State zinc-lead mining district embraces an extensive area, including the northeastern part of Ottawa County, Oklahoma, the southeastern part of Cherokee County, Kansas, and adjacent portions of Missouri, mainly in Jasper County. The Oklahoma-Kansas portion of the district, in the vicinity of Picher, Okla., has been the principal producing area since 1917, and the present milling practice in that section is the subject of this paper.

This district is the largest zinc-producing field in the United States. In 1926 it produced 55 per cent of the nation's zinc and 15 per cent of its lead, and in 1933 it produced 35 per cent of the zinc and 10 per cent of the lead. From 1842 to 1933, inclusive, the total output of the district was 18½ million tons of zinc and lead concentrates valued at 818 million dollars. Of this production, the Oklahoma-Kansas, or Picher, field yielded over 9 million tons valued at over 400 million dollars.

Mining operations in the Oklahoma-Kansas field near Picher and Baxter Springs began in 1915. The mills built at that time consisted merely of gravity concentration plants employing jigs and tables and the same flow sheets as had proved satisfactory with the free-milling ores such as were mined in the Joplin-Webb City fields in Missouri. The Kansas-Oklahoma ores proved to be much richer but also more refractory to treatment than those in the Joplin area, and contained a considerable percentage of zinc mineral not freed by the amount of crushing usually practiced in preparing the ores for jigging. Thus the old Joplin type of mill, with its usual two or four jigs and a few tables, proved inadequate and resulted in excessive tailings losses sometimes as high as 50 per cent of the mill feed. By employing more tabling equipment and regrinding jig middlings, locally termed "chats," the tailings losses were reduced somewhat, but the slime losses still accounted for 20 to 30 per cent of the mill-feed values.

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While flotation was practiced in a crude manner as early as 1915, it was not until 1924 that the recoveries by flotation became of real economic importance. Since 1924, flotation concentrate production has accounted for about 20 per cent of the total production.

Ten years ago relatively small tonnages were treated in each plant, 25 to 30 tons per hour being about the average. About that time plant capacities began to increase and by 1926 the average was close to 40 tons per hour, the increased tonnage being due in part to changes in flow sheets and coarser initial crushing. During the past few years the trend has been toward larger mills, so that capacities of 50 tons per hour are now common; several mills have capacities of 75 to 125 tons and the large Central mill of the Eagle-Picher Co., built in 1932, handles 225 tons per hour.

In former years the leasing system in vogue in the district resulted in many small operations, each landowner requiring that all ores mined on his land be milled separately in a mill built on his land. This caused the building of over 200 mills in the district by 1919, with capacities ranging from 20 to 50 tons per hour. On such a basis the initial cost of a plant had to be kept low, as it had to be amortized on the base of some 300,000 to 400,000 tons of crude ore mined. In 1926, the year of peak production of the district, the average number of mills operated throughout the year was 162, with a maximum of 183. In 1933, the number of mills operated ranged from about 10 to 40, and during the first six months of 1934, from 50 to 60 have operated, although about one-third of that number are tailings re-treatment plants.

The tendency in the district now is toward centralized milling; i.e., the treatment of ores from several leases over the same mill. This practice was initiated by the Bird Dog mill of the Commerce Mining and Royalty Co., which was put into operation in 1930. This plant demonstrated to the satisfaction of landowners, and in particular to the U.S. Department of the Interior in charge of restricted Indian lands, that fair and equitable settlements of royalties could be made on the basis of weighed and properly sampled mill feed. This, of course, can be done without presenting any problems new to the general practice of custom milling and smelting as practiced elsewhere. Centralized milling has been extended to tailings re-treatment plants also. Custom milling has developed with the centralized mills, which originally were devoted to milling ores of one company from its several leases.

As the existing central mills can produce over one-half the recent concentrate requirements of the district, and since other mills doubtless will be converted into central mills, it is very doubtful that much new mill construction will occur in the future.

As noted above, production of the district reached its peak in 1926, when a maximum of 183 mills operated with a maximum weekly output of over 19,000 tons of zinc concentrates and over 3000 tons of lead concentrates. This was followed by a gradual decline until the middle of 1929, when the sharp drop accompanying the business depression began and continued until the middle of 1932 when only 5 to 8 mills were operating and weekly production was less than 600 tons of zinc concentrates and 75 tons of lead concentrates.

It is doubtful whether the district can ever return to its 1926 basis of production, unless an unusual demand for its product should greatly stimulate future exploration and develop ore reserves now unknown. The high rate of depletion of orebodies may be visualized from the fact that in 1926, with an average of 160 mills operating, the equivalent of a 40-acre tract of mineral-bearing ground was worked out every two weeks.

Character of Ores Milled

A general knowledge of the ore occurrences is prerequisite to an understanding of the character of the ores milled, and the ore-dressing problems involved.

The ore deposits and the ore-bearing formations have been well described by various writers to whom the reader is referred for more detailed description. The ore-bearing formation is the Mississippian Boone limestone, which outcrops in the area embracing the old mining fields about Joplin and Webb City, Mo., and Galena, Kans., but is covered by later formations in the Picher area. Orebodies in the outcrop areas in the upper ground where weathering had occurred are locally called "soft-ground" deposits, and the others, such as occur throughout the Picher area, are called "hard-ground" deposits.

The Boone formation, essentially flat lying, was originally limestone but has been altered in the mineralized areas largely to chert, although in some places to dolomite. Where the beds locally have been more or less deformed, the chert was shattered and formed a "chert breccia" in which the fragments have been cemented by sphalerite and galena with or without contemporaneous black jasperoid.

In another type of deposit, the ore minerals appear stratified and are interbedded with thin chert bands or layers of chert nodules, which vary greatly in size. The ore minerals have replaced the layers of the formation that were unchertified or have been deposited along openings between the chert bands. Both the brecciated and stratified types of deposit are common throughout the Picher field, with which this paper is mainly concerned. The "sheet-ground" deposits, particularly notable in the old Webb City field, belong to the stratified type. In these, the bands of nearly pure sphalerite, with more or less galena, up to several inches in thickness and of great areal extent, are separated by layers of barren chert, or sometimes dolomite.

The several mineralized beds of thicknesses up to 20 to 25 ft. may be separated by beds of barren chert or limestone of considerable thickness. This, together with the irregular lateral distribution of the ore minerals throughout the ore-bearing beds, precludes selective mining in most instances and necessitates the mining of a large percentage of barren rock, thus diluting the zinc and lead values and necessitating milling methods that will permit very low operating costs in order to effect an economical separation of the very small percentage of ore minerals from the large proportion of barren chert. Because the ore minerals occur mainly as cementing material in the chert breccias or as layers or lenses between the chert bands or beds, a relatively coarse grind suffices to liberate most of them from the associated rock.

The ore minerals consist essentially of sphalerite and galena, fairly coarsely crystalline. The gangue material, aside from the barren rock, which is mostly chert, consists of jasperoid and also small amounts of calcite, dolomite, marcasite, pyrite and occasionally chalcopyrite and enargite. Aside from the chert and jasperoid, the gangue minerals do not usually offer any metallurgical problems on account of the very small proportion present. Occasionally, however, there is sufficient pyrite or calcite present to lower the grade of concentrate materially.

Ores of the breccia type containing abundant jasperoid, locally called "black rock," are most common throughout the Picher field. In much of the ore, the zinc and lead minerals are intimately mixed with the jasperoid. Such ores are locally referred to as "chatty" and are much more difficult to recover than when little jasperoid is present and the ore minerals are merely adhering to the chert. The latter ores are called "free-milling."

The free-milling ores offer no particular metallurgical problems other than those imposed by the economics of the situation. In milling such ores very low mill tailings with resultant high recoveries of the zinc-lead values are obtained under the usual practice of gravity concentration supplemented by flotation.

The chatty ores offer more difficult metallurgical problems, and generally cannot be made to yield as high recovery of the zinc values as do the free-milling ores. However, very satisfactory recoveries are being made, the limiting factors being the cost of plant operation and the condition of the metal market.

As noted above, the jasperoid was deposited as cementing material in fractures in the chert or in the breccias contemporaneously with the ore minerals. Thus part of the ore minerals occur disseminated throughout the jasperoid gangue in particles ranging from microscopic size up to several millimeters, the point of the unlocking of the sphalerite being generally about 48 mesh (Tyler scale). The portion of the zinc locked up in the jasperoid gangue presents at times very difficult ore-dressing problems in the isolation of so-called "chats" or middling grains into the so-called "chat circuit" or middling circuit, in order to permit the proper degree of grinding to unlock the zinc values. Separation of the chats from the barren chert and from the portion of the ore that is free-milling usually depends upon differences in specific gravity of less than one-tenth: hence jigging for a separation with such a small difference in gravity necessitates very close adjustments. Also, the physical shape of the "chats" or middlings resulting from crushing often makes the separation still more difficult.

The specific gravity of the chert gangue depends upon the extent to which the original limestone has been replaced by silica and the extent of subsequent leaching, if any, of included calcareous matter. The average specific gravity of the chert is about 2.60, but that of the porous cotton-rock phases may be as low as 2.35 and that of the most dense blue chert as high as 2.70.

TENOR OF ORES

Table 1 summarizes the operations of the entire district for the past six years and indicates the general tenor of the ores milled, as well as the magnitude of the industry.

The "concentration ratio" represents the number of tons of crude ore required to produce one ton of concentrate (tonnage of lead and zinc concentrates combined); generally this means that 15 to 16 tons of crude ore are required to produce one ton of concentrate. The term "extraction" is seldom used in this district, but the term "recovery," the reciprocal of the "ratio of concentration," is the commonly accepted expression. The average recovery of zinc concentrate has varied in the six years tabulated from about 4.97 to 8.48 per cent. The recovery of lead concentrate usually has been about one per cent or less. For this period of six years 29,431,800 tons of crude ore have been mined and milled with a production of 1,987,340 tons of zinc concentrates (average recovery 6.75 per cent) and 317,261 tons of lead concentrates (average recovery 1.08 per cent). It is evident that this is primarily a zinc-producing district.

The zinc and lead concentrates are sold on the basis of 60 per cent zinc and 80 per cent lead respectively. In the past, $40 per ton for zinc and $80 per ton for lead concentrates have been considered as normal prices for valuation purposes. During the first half of 1934, the prices have been about $28 for zinc and $43 for lead concentrates, but in 1932 were as low as $14 for zinc and $20 for lead concentrates.

Based on $28 for zinc concentrates and $43 for lead concentrates, and using 6.75 per cent as the average zinc recovery and 1.08 per cent as

TABLE 1.—*Tri-State Milling Operations*

Data from *Mineral Resources*, published by U.S. Bureau of Mines

Year	1928		1929		1930		1931		1932		1933	
Type of Operation	Mine	Tailing Mill	Mine	Tailing Mill	Mine	Tailing Mill	Mine	Tailing Mill	Mine	Tailing Mill	Mine	Tailing Mill
Tonnage Milled, short tons												
Oklahoma	5,527,700	2,500,300	6,408,400	2,916,200	4,118,600	3,095,000	2,208,100	1,620,800	1,262,200	325,500	2,188,200	1,433,900
Kansas	3,547,100	1,115,100	4,182,600	1,138,500	2,355,100	1,162,200	1,040,800	872,400	507,100	243,400	725,400	503,600
S. W. Missouri	281,300		428,400		328,800		104,800		46,400		170,800	
District total	9,356,100	3,615,400	11,019,400	4,054,700	6,802,500	4,257,200	3,353,700	2,493,200	1,815,700	568,900	3,084,400	1,937,500
Metal Content Recovered, per cent												
Zinc:Oklahoma	3.21	0.88	3.11	0.85	3.08	0.99	3.94	1.07	3.66	1.37	3.63	0.97
Kansas	3.09	0.93	2.93	0.82	3.23	0.73	3.80	1.24	4.24	1.17	4.03	0.91
S. W. Missouri	3.23		2.26		2.25		2.15		2.66		2.51	
District average	3.17	0.90	3.01	0.84	3.09	0.92	3.84	1.13	3.80	1.28	3.65	0.95
Lead: Oklahoma	0.69	0.01	0.62	0.01	0.71	0.02	0.68	0.01	0.63	0.04	0.72	
Kansas	0.72	0.01	0.63	0.01	0.65	0.01	0.73	0.03	0.72		0.63	
S. W. Missouri	0.41		0.25		0.31		1.15		2.48		0.60	
District average	0.69	0.01	0.61	0.01	0.67	0.02	0.71	0.02	0.70	0.04	0.70	0.02
Zinc and lead combined, district average	3.86	0.91	3.62	0.85	3.76	0.94	4.55	1.15	4.50	1.32	4.33	0.97
Concentrates Shipped,[a] short tons												
Zinc: Oklahoma	315,717	38,354	340,380	45,697	215,821	52,350	123,680	24,432	108,332	9,944	148,659	23,554
Kansas	186,113	18,219	193,916	16,337	128,367	14,981	55,537	18,153	44,168	5,319	69,412	7,834
S. W. Missouri	17,397		13,308		11,172		3,600		1,538		10,123	
District total	519,227	56,573	547,604	62,034	355,360	67,331	182,817	42,585	154,038	15,263	228,294	31,388
Lead Oklahoma	56,449	390	50,666	375	38,054	946	16,793	212	13,437	212	23,193	445
Kansas	32,259	158	34,143	209	19,758	165	8,882	401	8,375	4	7,814	18
S. W. Missouri	1,413		1,263		1,192		1,254		849		1,477	
District total	90,121	548	86,072	584	59,004	1,111	26,929	613	22,661	216	32,474	463
Combined zinc and lead, total	609,348	57,121	633,676	62,618	414,364	68,442	209,746	43,198	176,699	15,479	260,768	31,851
Ratio of Concentrates to Mill Feed, per cent, i.e., recovery												
Zinc	5.55	1.56	4.97	1.53	5.22	1.58	5.45	1.71	8.48	2.68	7.76	1.62
Lead	0.96	0.02	0.78	0.01	0.87	0.03	0.80	0.02	1.25	0.04	1.12	0.02
Combined zinc and lead	6.51	1.58	5.75	1.54	6.09	1.61	6.25	1.73	9.73	2.72	8.88	1.64
Average Metal Content, per cent												
Zinc concentrates, zinc	58.6	56.9	59.0	56.5	59.0	58.0	60.3	59.6	60.8	60.0	60.5	58.7
Lead concentrates, lead	78.8	69.5	78.3	63.7	77.4	62.6	79.1	66.7	79.3	73.1	78.3	63.8
Average Value per Ton Concentrates												
Zinc	$37.26	33.16	39.80	35.33	31.31	27.91	23.94	21.03	18.37	17.22	25.97	27.37
Lead	$80.50	70.15	84.75	66.64	65.96	48.29	45.57	33.59	36.07	26.56	44.45	27.95
Weighted combined zinc and lead	$43.66	33.51	45.90	35.62	36.24	28.24	26.72	21.21	20.64	17.35	28.24	27.38

[a] These figures show concentrates shipped during each calendar year rather than concentrates produced from the indicated tonnages milled, since production figures are really only estimates until final shipments are made. Obviously, recoveries figured on shipments are slightly incorrect.

the average lead recovery, the average grade of ore would yield $2.35 as the gross recoverable values of the mill heads in this district. It is obvious that only a very small margin of profit is possible at prices of $30 or less for zinc concentrate, necessitating a proper balancing of operating cost against metal recovery in order to realize the greatest net profit. At low prices, the higher grade ores must be mined and mill tailings adjusted to permit the best over-all profit to the producers.

GENERAL MILLING PRACTICE

The foundation of milling practice in the Tri-State district is gravity concentration supplemented by flotation. Numerous reasons support this practice, the principal one being the character of the ores.

Jigging, as developed in this district, serves well in discarding economic tailings from an unsized feed with maximum size of particles from ⅜ to ⅝ in. As an adjunct to the jigs for recovering granular mineral not recovered in the jigging department and that resulting from the regrind of jig middlings, tables are generally used. The slimes produced in mining operations and from the crushing and grinding in the gravity-concentration department are recovered by flotation.

The flow sheet groups the milling steps under four major headings: (1) ore preparation; (2) primary gravity concentration; (3) secondary gravity concentration, and (4) slime concentration.

1. *Ore Preparation.*—The mine run of ore after the boulders of waste have been hand-culled is ground to a size ranging from minus ⅜ to ⅝ in., by a jaw crusher and rolls. The crushing usually is done wet, but in some of the newer plants is done dry, all or in part.

2. *Primary Gravity Concentration.*—After removal of the slimes the ore is jigged, which produces an enriched product for recleaning, a middling product for regrind and re-treatment and an economic tailing for discard. The enriched product is rejigged for the separation of the lead and zinc and the production of finished concentrates. The tailing from the cleaner jig is not wasted but reground and re-treated by secondary gravity concentration.

3. *Secondary Gravity Concentration.*—The middlings from the jigging operations, or the primary stage of gravity concentration, are reground and re-treated in the secondary stage of gravity concentration by both jigs and tables, or tables only. Practice varies considerably in this stage of milling. Finished concentrates are produced and economic tailings are discarded.

4. *Slime Concentration.*—All slimes resulting from mining operations, those produced by the initial crushing of the ore to jig size and those produced from the regrind of all gravity middling produced go to the flotation department for recovery of the slime mineral.

While no two flow sheets are alike, they all fall under this general scheme of ore dressing, except that in a few cases secondary gravity concentration by tables is not used.

While the character of the ores milled is the primary reason for the milling practice adopted, another important factor is the relatively short life of operations for any one plant, necessitating a high amortization rate for plant. The criteria, therefore, for concentrator construction and flow sheet in this district as pertains to the usual operation are: (1) a low operating cost; (2) a low plant investment, and (3) flexibility of flow sheet and tonnage capacity to permit of operations over a wide range of prices for concentrates produced.

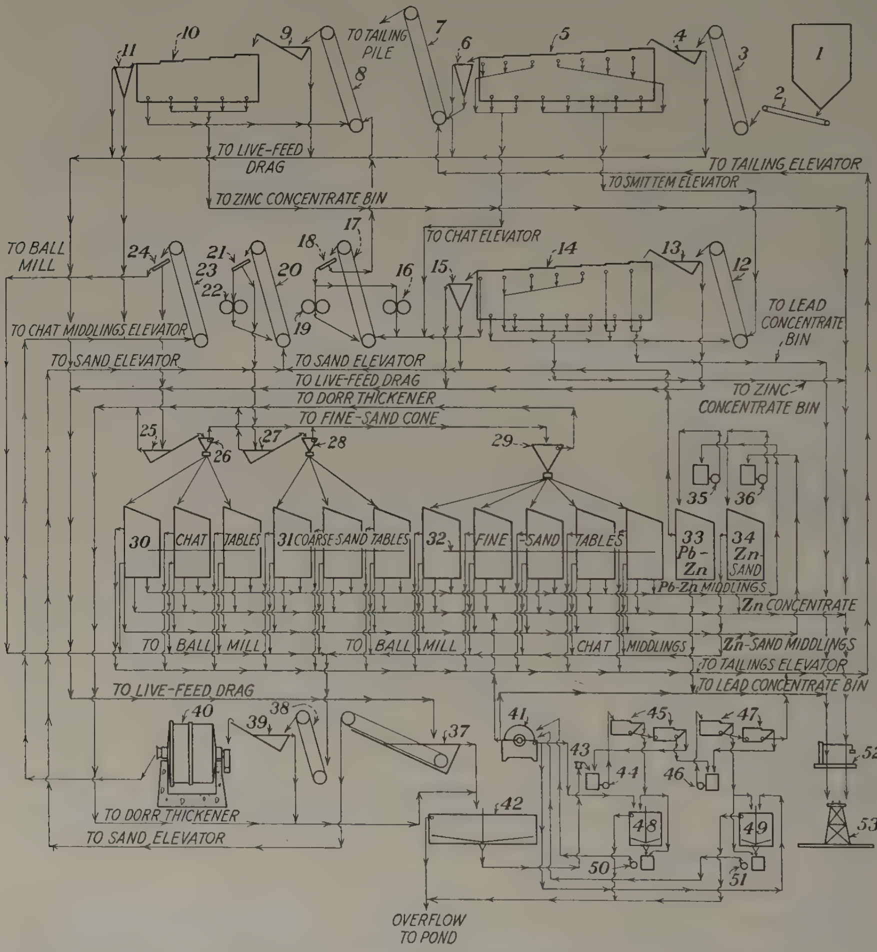


Fig. 1.—Concentrator flow sheet, Ballard Mill, St. Louis Smelting and Refining Co. Legend on opposite page.

The flow sheets given in Figs. 1, 2, 3 and 4 are typical of the largest and most recently erected concentrators in the district, all having been built within the past five years.

DETAILED MILLING PRACTICE

Ore Storage and Conveyance

Usually the mill is located so that the ore as mined can be hoisted from a shaft, designated as the "mill shaft," and dumped directly into the mill feed bin. When mining operations are carried on in more than the mill shaft, the other shafts are called "field shafts." Each field shaft has a storage bin of from 100 to 200 tons capacity. A surface haulage system, together with an incline tram, serves to convey the ore from the respective field shafts to the mill storage bin, which has a capacity of from 200 to 400 tons.

FIG. 1.—CONCENTRATOR FLOW SHEET, BALLARD MILL, ST. LOUIS SMELTING AND REFINING CO., MIAMI, OKLAHOMA.

1. 1000-ton circular steel fine-ore bin.
2. 30-in. belt conveyor driven by variable-speed motor.
3. 30-in. belt elevator (dirt elevator).
4. Desliming drag, 30-in. belt, 5 ft. wide (dirt drag).
5. Two 42 by 48-in. seven-cell steel rougher jigs.
6. Demier dewatering cone, 3 by 5 by 5 ft.
7. Tailing elevator, 30-in. belt.
8. Chat-jig elevator, 24-in. belt.
9. Chat-jig drag, 20-in. belt.
10. Chat jig, five-cell, 36 by 48 in., steel.
11. Demier dewatering cone, 3 by 5 by 5 ft.
12. Smittem elevator, 24-in. belt.
13. Smittem drag, 20-in. belt.
14. Cleaner jig, seven-cell, 36 by 48-in., steel.
15. Demier dewatering cone, 3 by 5 by 5 ft.
16. Chat rolls, 42 by 16-in. W.C.C.F. & M. Co., high-speed.
17. Chat regrind elevator, 24-in. belt.
18. Two chat screens, 4 by 5-ft. Leahy.
19. Chat rolls, 42 by 16-in. W.C.C.F. & M. Co., high-speed.
20. Sand elevator, 24-in. belt.
21. Two sand screens, 4 by 5-ft. Leahy.
22. Sand rolls, 42 by 16-in. W.C.C.F. & M. Co., high-speed.
23. Chat middling elevator, 24-in. belt.
24. Two chat middling screens, 4 by 5-ft. Leahy.
25. Chat middling drag, 20-in. belt, 4 ft. wide.
26. Chat middling-classifying cone, 24-in. diameter.
27. Sand drag, 20-in. belt., 4 ft. wide.
28. Sand-classifying cone, 24-in. diameter.
29. Fine-sand classifying cone, 42-in. diameter.
30. Three Butchart concentrating tables (chat).
31. Three Butchart concentrating tables (coarse sand)
32. Five Butchart concentrating tables (fine sand).
33. One Butchart concentrating table (Pb-Zn mid.).
34. One Butchart concentrating table (Zn-sand mid.)
35. 2-in. Wilfley pump (Pb-Zn mid.).
36. 2-in. Wilfley pump (Zn-sand mid.).
37. Live-feed drag, 20-in. belt, 16 ft. wide.
38. Ball-mill elevator, 24-in. belt.
39. Ball-mill drag, 20-in. belt, 4 ft. wide.
40. Marcy ball mill, 64½.
41. American concentrate filter, five-disk, 6-ft. diameter.
42 Butchart slime thickener, 60-ft. diameter.
43. Butchart 4-in. diaphragm pump.
44. Wilfley 2-in. pump, Pb pulp.
45. Two Butchart Pb flotation machines.
46. Wilfley 2-in. pump, Zn pulp.
47. Two Butchart Zn flotation machines.
48. Pb concentrate thickener, 16-ft. dia. by 10-ft stave.
49. Zn concentrate thickener, 16-ft. dia. by 10-ft. stave.
50. Pb concentrate pump.
51 Zn concentrate pump.
52. Scales.
53. Ore bin.

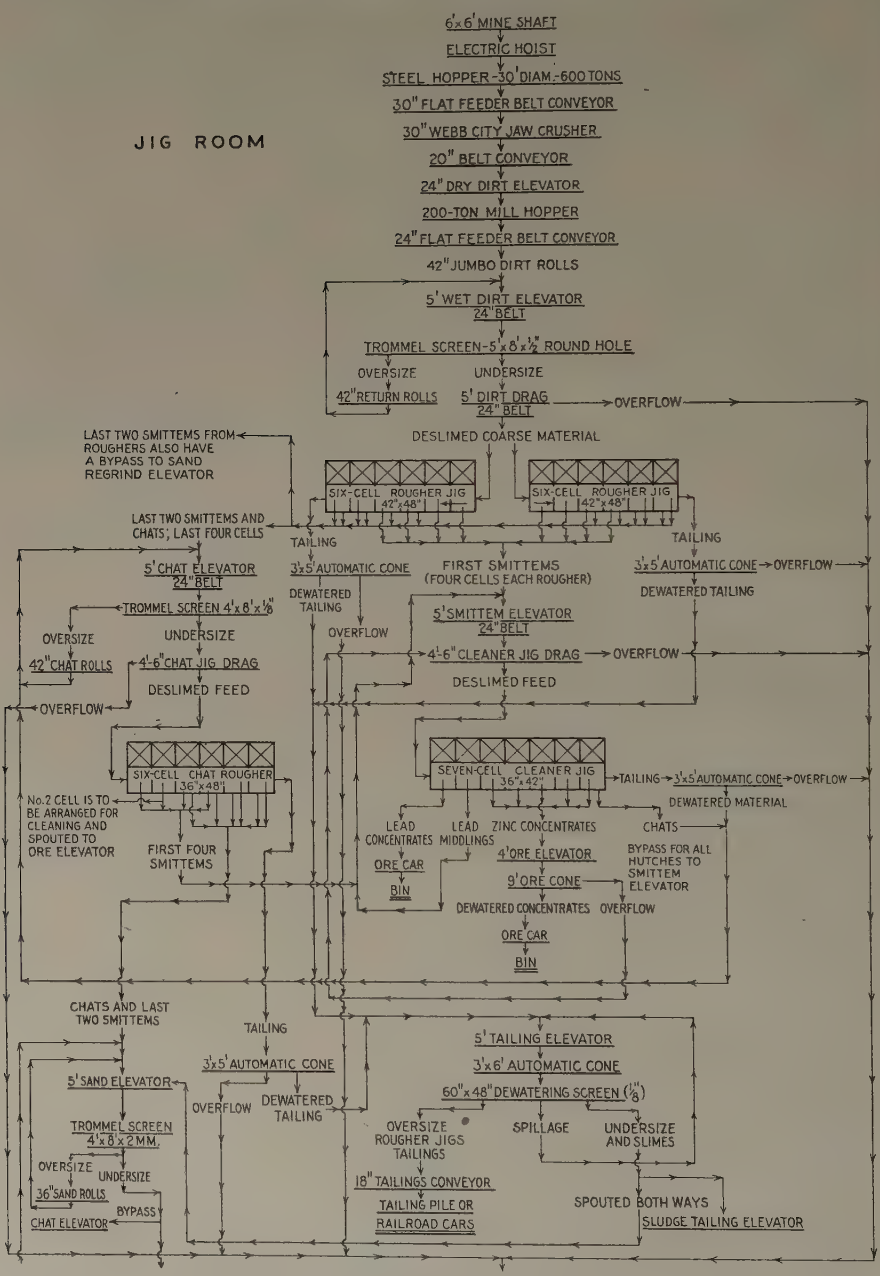


FIG. 2.—FLOW SHEET OF SCOTT MINE, CENTURY ZINC CO.

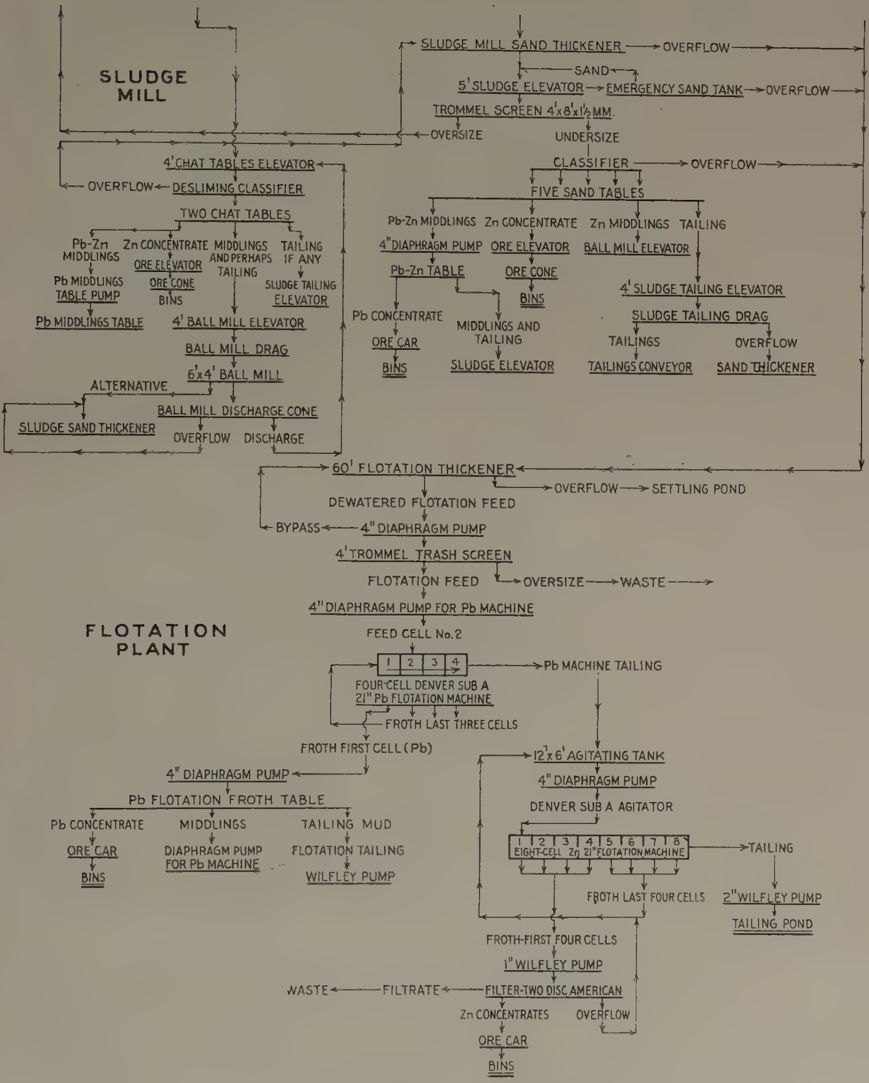
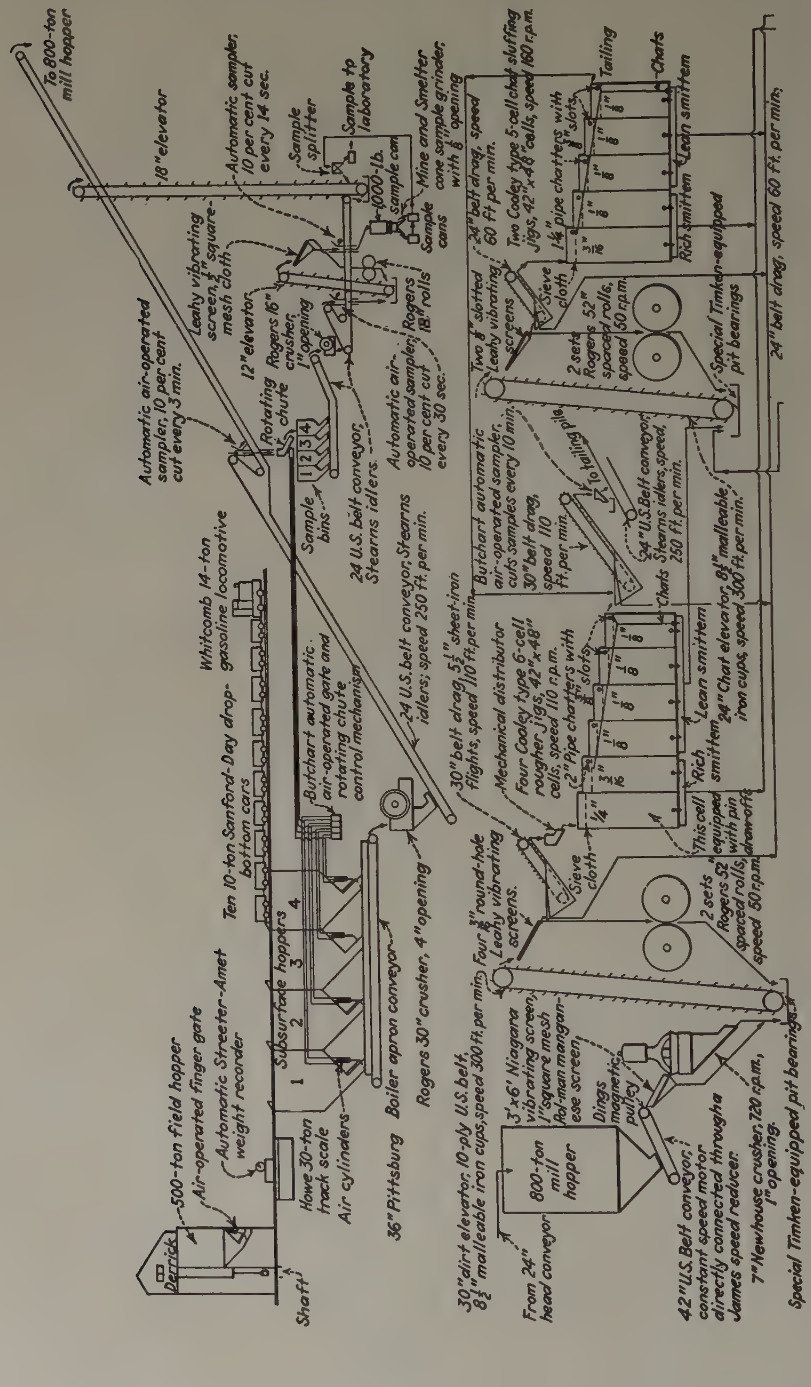


FIG. 2.—(CONTINUED.)


To 800-ton mill hopper
18" elevator
Automatic sampler, 10 per cent cut every 14 sec.
Sample splitter
Sample to laboratory
1000-lb. sample can
Mine and Smelter cone sample grinder, with 1/8" opening
Sample cans
Leahy vibrating screen, 1/2" square-mesh cloth
12" elevator
Rogers 16" crusher, 1" opening
Rogers 18" rolls
Automatic air-operated sampler, 10 per cent cut every 30 sec.
Automatic air-operated sampler, 10 per cent cut every 3 min.
Rotating chute
1 2 3 4
Sample bins
24 U.S. belt conveyor, Stearns idlers.
Whitcomb 14-ton gasoline locomotive
24 U.S. belt conveyor, Stearns idlers; speed 250 ft. per min.
Ten 10-ton Sanford-Day drop-bottom cars
Butchart automatic air-operated gate and rotating chute control mechanism
Subsurface hoppers
1 2 3 4
500-ton field hopper
Air-operated finger gate
Automatic Streeter-Amet weight recorder
Derrick
Shaft
Howe 30-ton track scale
Air cylinders
36" Pittsburg Boiler apron conveyor
Rogers 30" crusher, 4" opening
30" belt drag, 5 1/2" sheet-iron flights, speed 110 ft. per min.
Mechanical distributor
Four Cooley type 6-cell rougher jigs, 42"x48" cells, speed 110 r.p.m. (2" Pipe chatters with 3/8" slots)
1/4" 3/16" 1/8" 1/8" 1/8" 1/8"
Chats
Rich smittem
Lean smittem
This cell equipped with pin draw-offs
24" Chat elevator, 8 1/2" malleable iron cups, speed 300 ft. per min.
Sieve cloth
2 sets Rogers 52" spaced rolls, speed 50 r.p.m.
Four 3/8" round-hole Leahy vibrating screens.
30" dirt elevator, 10-ply U.S. belt, 8 1/2" malleable iron cups, speed 300 ft. per min.
3'x6' Niagara vibrating screen, 1" square mesh Rol-man manganese screen
Dings magnetic pulley
800-ton mill hopper
From 24" head conveyor
42" U.S. Belt conveyor, constant speed motor directly connected through a James speed reducer.
7" Newhouse crusher, 720 r.p.m., 1" opening.
Special Timken-equipped pit bearings
Butchart automatic air-operated sampler, cuts samples every 10 min.
30" belt drag, speed 110 ft. per min.
To tailing pile
24" U.S. Belt conveyor, Stearns idlers, speed 250 ft. per min.
Two 1/8" slotted Leahy vibrating screens
Sieve cloth
2 sets Rogers 52" spaced rolls, speed 50 r.p.m.
Special Timken-equipped pit bearings
24" belt drag, speed 60 ft per min.
Two Cooley type 5-cell chat sluffing jigs, 42"x48" cells, speed 160 r.p.m.
1 1/4" pipe chatters with 1/8" slots
3/16" 1/8" 1/8" 1/8" 1/8"
Tailing
Chats
Rich smittem
Lean smittem
24" belt drag, speed 60 ft. per min.

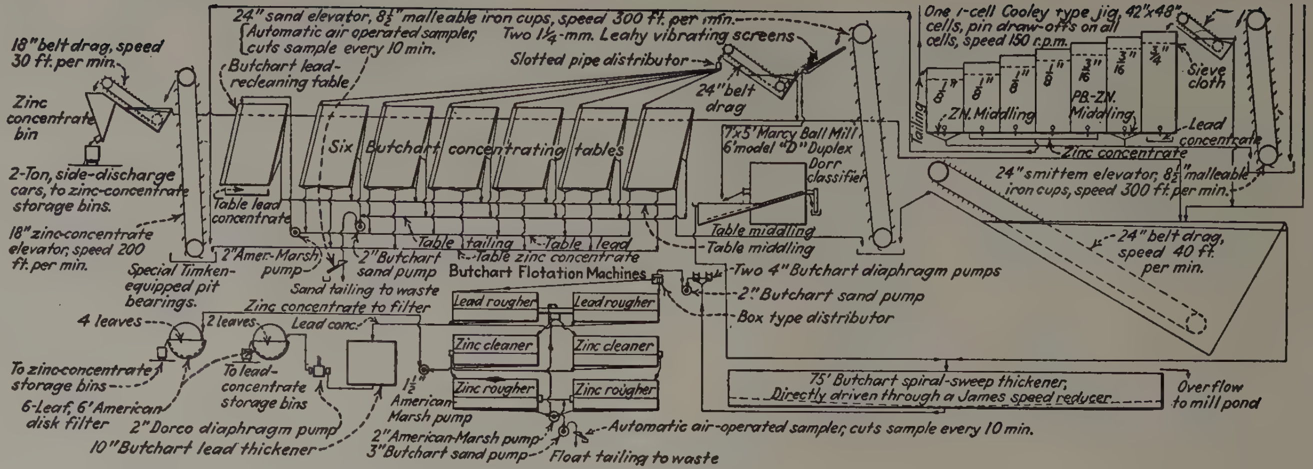

18" belt drag, speed 30 ft. per min.
Zinc concentrate bin
2-Ton, side-discharge cars, to zinc-concentrate storage bins.
18" zinc-concentrate elevator, speed 200 ft. per min.
Special Timken-equipped pit bearings.
24" sand elevator, 8½" malleable iron cups, speed 300 ft. per min.
Automatic air operated sampler, cuts sample every 10 min.
Butchart lead-recleaning table
Two 1¼-mm. Leahy vibrating screens
Slotted pipe distributor
24" belt drag
Six Butchart concentrating tables
Table lead concentrate
2" Amer-Marsh pump
2" Butchart sand pump
Sand tailing to waste
Table tailing
Table lead
Table zinc concentrate
Butchart Flotation Machines
Zinc concentrate to filter
Lead conc.
4 leaves
2 leaves
To zinc-concentrate storage bins
6-Leaf, 6' American disk filter
To lead-concentrate storage bins
2" Dorco diaphragm pump
10" Butchart lead thickener
1½" American Marsh pump
2" American-Marsh pump
3" Butchart sand pump
Lead rougher
Lead rougher
Zinc cleaner
Zinc cleaner
Zinc rougher
Zinc rougher
Float tailing to waste
Automatic air-operated sampler, cuts sample every 10 min.
7'x5' Marcy Ball Mill
6' model "D" Duplex Dorr classifier
Table middling
Table middling
Two 4" Butchart diaphragm pumps
2" Butchart sand pump
Box type distributor
75' Butchart spiral-sweep thickener, Directly driven through a James speed reducer
Overflow to mill pond
24" belt drag, speed 40 ft. per min.
24" smittem elevator, 8½" malleable iron cups, speed 300 ft. per min.
One 7-cell Cooley type jig, 42"x48" cells, pin draw-offs on all cells, speed 150 r.p.m.
1/8"
1/8"
1/8"
1/8"
3/16"
3/16"
3/4"
Sieve cloth
Tailing
ZN. Middling
PB-ZN. Middling
Lead concentrate
Zinc concentrate

FIG. 3.—FLOW SHEET OF BIRD DOG MILL. (*Reprinted from Eng. & Min. Jnl.*, Jan. 26, 1931.)

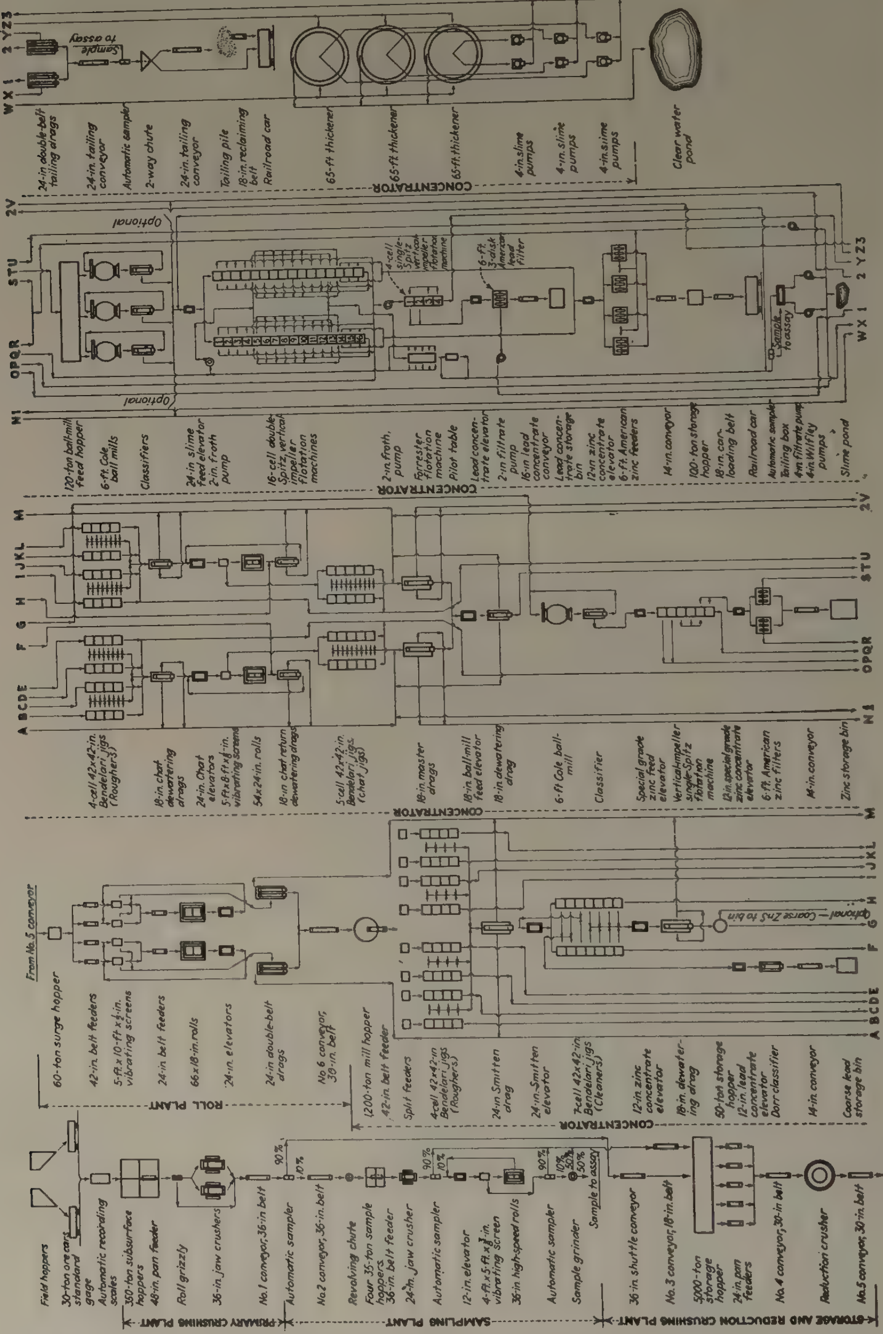


FIG. 4.—FLOW SHEET OF EAGLE-PICHER CENTRAL MILLING PLANT. (*Reprinted from Eng. & Min. Jnl.*, May, 1932.)

The surface haulage system usually consists of a train of 2 to 3-ton end-dump tram cars and a 3 to 5-ton locomotive, both steam and gasoline types of locomotive being employed, with the latter predominating in recent years.

In one or two instances all the ore is hoisted through the mill shaft and dumped directly into the mill bin, the ore from the field shafts being trammed underground to the mill shaft.

It is the general practice to dump all ore as hoisted on to bar grizzlies with openings of from 6 to 8 in., to hand-cull and discard the boulders of waste rock and break up the boulders of ore by hand with 8-lb. sledge hammers. The men doing this work are known locally as "screen apes."

Crushing and Screening

After passing through the bar grizzly into the mill bin, the ore generally is hand-fed through a gate and chute directly to a jaw crusher. No screen or grizzly is employed to screen out the undersize from the feed to the crusher.

Crushing, with a few exceptions, is done wet, water being added at the crusher. The jaw crushers used range in size from a width of mouth of 16 to 36 in. and are set with a minimum opening of from 1 to 2 in. The local practice is to use a short crusher stroke of from ¾ to ⅞ in. and to run at high speed, from 300 to 350 r.p.m. The chert rock, while very abrasive, is extremely brittle and breaks satisfactorily with a short stroke. The high speed makes possible the crushing of unusually high tonnages in comparatively small crushers.

The product of the jaw crusher passes through a spout underneath the crusher directly to the primary crushing rolls, locally called "dirt rolls." These rolls range in size from 16 in. face by 36-in. dia. up to 24-in. face by 54-in. dia., the usual size being 16-in. face by 42-in. dia. In the majority of cases slow-speed geared Cornish rolls are used, the speed of the rolls varying from 28 to 38 r.p.m. The larger rolls, such as 24-in. face by 54-in. dia., are of the spaced type and run from 45 to 65 r.p.m. The reduction accomplished in the primary or "dirt rolls" is usually from 1½ to 2-in. material down to ¾ to 1-in. material. At this stage of the crushing, generally speaking, 40 to 50 per cent of the product from the "dirt rolls" passes through a ½-in. round-hole screen, 60 to 70 per cent of the entire product is minus ¾ in., and the oversize on ¾ in. ranges in size up to 1½ in. maximum one-way dimension.

In one or two instances, trommel screens with ¾-in. round punched plate are employed to remove the undersize from the feed to the dirt rolls, and for rich ore, especially high in lead, such practice is justified on the basis of the increased mineral production from the jigging department. For the usual grade of ore milled, it is doubtful whether such a procedure is justified.

The product of the dirt rolls passes by gravity to the so-called "dirt elevator," the belt of which ranges in width from 18 to 30 in. This elevator discharges to the so-called "dirt" screen or screens, consisting usually of 4 by 8-ft. trommels with punched plate for screen jackets, the size of hole ranging from ⅜ to ⅝ in., the average being about ½ in. The oversize from the dirt screens passes by gravity to regrind rolls, locally termed the "return rolls." These rolls are of the slow-speed geared Cornish type, ranging in size from 16-in. face by 36-in. dia. to 16-in. face by 42-in. dia. The speeds range from 28 to 38 r.p.m. The product of the return rolls passes by gravity to the dirt elevator, the latter, together with the dirt screen and return rolls, being in closed circuit.

The undersize from the dirt screen passes directly, without any intermediate storage or surge bins, to the dewatering and desliming equipment at the head of the jigging department.

Rougher Jigging

The dirt-screen product, after desliming, makes up the feed to the rougher jigs, which, together with the preceding desliming, effect the primary concentration of the mill-feed values into products for subsequent re-treatment and discard from 60 to 80 per cent of the tonnage as an economic tailing.

The desliming equipment consists of either a drag classifier of the Esperanza type or automatic cones of the Boylan, DeMier or Wood types. While the operation of the cones is satisfactory, represents a lower initial cost and does not require any power, the cones require considerably more headroom than the classifier and do not give as regular a feed to the jigs. While the home-made drags are not as efficient in the removal of fines and slimes as the factory-made types, such as the Dorr and Akins, their lower initial cost makes them popular.

The rougher jigs, as operated locally, do not make a satisfactory recovery of the minus 65-mesh zinc mineral, therefore the primary concentration of the minus 65-mesh mineral from the mill feed depends upon efficient desliming ahead and following the rougher jigs. By use of the Esperanza type of drag classifier, it is possible to deslime the rougher-jig feed so as to show only about 2 per cent of minus 65-mesh plus 200-mesh material.

Screen analysis of the overflow from the desliming equipment should show very little, if any, plus 35-mesh material. This overflow passes either to sand tanks, sand cones, sand thickeners or large sand drags, known locally as "live-feed" drags, where the sand is separated from the slime. This sand goes to make up in part the feed to the table department and the slime goes to slime thickeners for flotation.

Generally 4 to 6 per cent of the mill-feed tonnage is contained in the overflows from the desliming equipment in the jig department, representing 5 to 8 per cent of the mill-feed values, and represents the primary concentration of the minus 65-mesh slimes, their subsequent recovery depending upon secondary concentration in the table and flotation departments. The object, therefore, of the rougher jigs is to effect the recovery of as much as is economically possible of the plus 65-mesh values from a mixed or unsized feed that has been thoroughly deslimed. Sizing of the feed to the rougher jigs is not practiced, and the feed ranges from plus 65-mesh to ⅝ inch.

The rougher jigs produce a rough concentrate locally called "smittem" for recleaning on the cleaner jig without further regrinding, a middling product, locally termed "chats," requiring regrinding for liberation of the contained values, and a tailing for discard. The smittem generally contains from 15 to 30 per cent of the feed to the rougher jig and assays from 25 to 30 per cent zinc with varying percentages of lead, the tonnage and values being dependent primarily upon the richness of the rougher jig feed. The chat middling product usually contains from 5 to 10 per cent of the rougher-jig feed and assays from 4 to 8 per cent zinc, the tonnage being dependent primarily upon the character of ore milled; i.e., whether chatty or free-milling.

The jig tailing represents from 60 to 80 per cent of the rougher-jig feed and assays from 0.75 to 1.00 per cent zinc in mills doing what is economically possible under the proper mill supervision and operation. However, rougher-jig tailings considerably higher than these figures are being produced in mills that are not subject to proper supervision. The percentage of tailings discarded depends primarily upon the character of ores being milled, the chatty ores necessitating the drawing of a larger percentage of chats or middlings than the free-milling ores. The values in the tailings also depend upon the character of ores milled.

To sum up, the smittem will contain about 80 to 85 per cent of the values in the rougher-jig feed, the chats 5 to 10 per cent, and the tailings 10 to 15 per cent. In other words, the rougher jig effects a primary concentration of 85 to 90 per cent of the values contained in its feed into products for retreatment amounting to 20 to 40 per cent of the tonnage going to the rougher jig. The extraction of the free mineral coarser than 65 mesh is excellent, usually about 95 per cent being recovered. With decreasing size of the gangue the rejection decreases, and a very large percentage of the minus 2-mm. sand appears in the hutches and must be rejected by subsequent treatment. It is obvious, therefore, that the rougher jig is the key not only to the over-all metallurgical recovery effected, but to the milling costs obtained.

The rougher-jig tailing must be an economical product, in that a lowering of its values must justify the cost of treating the increased tonnage of chats or middlings produced thereby. This means that fluctuations in the metal market should govern, to a certain degree, what can be considered as the most economical rougher jig tailing that should be produced.

Two types of jigs are now in use in the field, both of which perform in a commendable manner. The Cooley jig, which is a modification of the Harz type, has long been the standard, and predominates. The Bendelari diaphragm type is now being used exclusively in the new Eagle-Picher central mill.

With the Cooley jig, six cells, 42 in. wide by 48 in. long, in series, are usually employed, the fall between cells generally being 3 in. In design, the plunger compartment is somewhat larger than the sieve cell, so that the ratio of areas is a little over unity, the common practice being to make the former 4 in. wider than the latter. The ratio of throat area to sieve is less than unity, the usual practice being to make the throat when measured at right angles to the pitch boards, two-thirds the width of the sieve. The pitch board of the last cell is usually given a minimum pitch of 8 in. in 12, and the pitch boards for the other cells depend upon maintaining the same throat opening for all cells. The depth of the partition between the plunger and sieve compartments is usually about four-tenths of the width of the sieve cell. The height of the tail board above the sieve level ranges from 6 to 8 in., the former being more common. Hydraulic water is usually added from open launders above the plunger and passes between the plunger and the sides of the plunger compartment into the hutch compartment. In some plants the water is now added, either under or above the plungers, by individual pipe lines from a header behind the jig, thus giving more positive regulation of the plunger water.

The Bendelari diaphragm jig is constructed of steel plate. It has a round plunger directly beneath the jig sieve and the jig cell and hutch compartment are square. The plunger rod is fastened rigidly to the under side of the plunger and is connected (by means of a crosshead and connecting rod) to an eccentric also located under the plunger. The plunger water is prevented from escaping through the opening required for the plunger rod by means of a large diaphragm bolted to the outer circle of the plunger. Provision is made to allow the hutch product to pass by the plunger at the four corners of the hutch compartment. This jig, of course, occupies the minimum of space.

While many factors enter into the efficient operation of the rougher jig, no particular difficulties are encountered in efficiently extracting the plus 65-mesh free mineral. The size of the sieve opening in each respective cell, the total depth of material on the sieve, the depth and character of the bedding on the different cells, and the pulsion velocity imparted to each cell by the plunger are all governing factors. While each of these may vary through an appreciable range, they must all be balanced in many combinations in order to produce satisfactory results. The speed of the rougher jigs varies from 90 to 120 strokes per minute, with corresponding variations in the length of strokes from 2 in. down to 1 in. Very little, if any, difference is to be noted, though, in the performance of the jigs by varying the speed between these limits when the length of the stroke is properly adjusted to each respective speed so as to give the proper pulsion velocity. One of the most vital factors is the amount of plunger water used, as an increase of plunger water decreases suction, and vice versa. While sufficient plunger water is required to soften the whole bed of material on the sieve and permit the ready settlement of the coarse mineral through the bed with the minimum number of pulsions, sufficient suction has to be maintained in order to effect a satisfactory recovery of the fines by sucking them through the interstices of the coarse particles. In other words, when jigging mixed or unsized material, both suction and pulsion are required and the proper balancing of the two opposing forces results in the most efficient jigging.

A six-cell 42 by 48-in. rougher jig generally handles from 25 to 30 tons per hour of mill feed from which have been removed fine sand and slimes, and produces an economical tailing. The accumulation of top water, however, makes the recovery of the light chat or middlings on the last two or three cells more difficult. With the view of overcoming this, different arrangements have been tried, such as feeding a six-cell jig in the middle and dividing the feed to three cells each way, or splitting the feed into as many parts as there are cells, giving the equivalent of single-cell operation. To date, series operation is the most popular and beyond doubt is as effective as any other arrangement. However, when a proper correlation is made between milling mine ore and re-treating tailings, it appears that the proper procedure would be to employ one four-cell 42 by 48-in. rougher for the recovery of free mineral only (and on such a basis a jig duty of 25 to 30 tons per hour is indicated), then to dewater the jig tailings and split to two three-cell 42 by 48-in. chat roughers for the recovery of the middlings. In other words, in the re-treatment of tailings it has been found economically advisable to rejig at about one-half the tonnage rate for the recovery of the chats lost in the original operations.

One man handles the operation of two six-cell rougher jigs. The jig operator shovels the free lead and zinc minerals that accumulate on top of the sieve into the spout going to the cleaner jig. Usually the first four cells suffice to extract the free mineral and the contents of these hutches go to the cleaner jig. The contents of the last two hutches, together with the chat or middling draw, go to the chat or middling circuit for regrinding and secondary concentration, as will be discussed later. Frequent cleaning or "spudding" of the sieve cloths is necessary to keep the sieves from blinding. This is done with a dull-edged chisel bar called locally a "spud." Sometimes a small air hammer is used on the end of the spud in order to jar the sieve and assist in cleaning it.

The method used for removing the middlings or chats on the last two or three cells is not as efficient as desired. The problem is difficult. Much time and patience have been applied in this direction and, needless to say, much more must be given before the problem will be satisfactorily solved. To insure as complete a removal as possible of these chats, so-called "chatters" are installed on the last three cells. Many designs have come and gone but the pipe type seems to have stayed. It consists of a 2-in. pipe with ¾-in. holes drilled in it on 4-in. centers, or a long slot instead of the holes, positioned at the tail end of the cell and parallel to the tail board. In elevation the holes or slot are arranged to be at the horizon of the settled chats and facing the direction of feed. The back end of the pipe is fitted with an elbow directed upward through which water may be added to assist in washing the trapped chats to the outside of the jig wall. Many chats are trapped but much barren gangue is caught also, making the middling draw much leaner in value than desired. The average specific gravity of the chert is about 2.60, and the specific gravity of chats ranges from 2.6 to 4.0. The stratification of these "chats" is fairly well accomplished in the gravities above 2.74, which assay over 10 per cent zinc.

The tailings from the rougher jigs are dewatered and further deslimed in either automatic cones or drag-type classifiers and disposed of by either tailing elevators or belt conveyors.

Cleaner Jigging

The hutch products (except from the last two or three cells) and the bed material from the first three or four cells of the rougher jigs, make up the so-called "smittem," or feed to the cleaner jig. It is laundered to the smittem elevator and elevated to some type of desliming device such as an automatic cone or Esperanza-type drag classifier, which in turn feeds the cleaner jig, the overflow or slime joining the overflows from the desliming equipment serving the rougher jigs.

The cleaner jig is much like the rougher in construction details, differing only in the number and size of cells and the fall per cell. Generally it consists of seven cells 36 by 42 in. with a 1½-in. fall per cell, although some prefer 36 by 48-in. cells. The speed of the cleaner jig is almost without exception from 160 to 165 r.p.m., the strokes varying from 1¾ in. on the first cell down to ¾ in. on the last cell.

Generally, the first cell suffices for a recovery of the lead and makes a finished lead concentrate. In some cases, however, when the lead content is higher than usual, it is necessary to use the first two cells. In either instance the succeeding cell produces a lead-zinc middling, which is returned to the smittem elevator. The remaining cells are devoted to the production of finished zinc concentrates, with the last cell at times making a middling product that is returned to the smittem elevator.

The tailing from the cleaner jig is a middling product and all of it receives further treatment, as discussed later.

The beds on the cleaner are kept deep enough to produce a finished concentrate in the hutch. It is the usual practice to use pin draws on both the head lead cells and the head zinc cells. The pins consist of ¾-in. round iron bars which fit into holes punched in the sieve cloth. When the accumulated coarse concentrates have filled the top side of the cell, these pins are withdrawn and the coarse mineral allowed to drop into the hutch. Some plants prefer the usual gate and dam bed draw, or the local type of chatter, to draw off the coarse mineral, and others prefer to shovel off the coarse mineral. All these methods are satisfactory.

The hutch draws are arranged so that if trouble occurs in operation the hutch may be drained and the contents returned to the smittem elevator and cleaned again. The operation of the cleaner jig requires the attendance of one man known as the "cleaner man."

The whole object of this operation is to separate the lead and zinc into respective finished concentrates, the grade of each being that which represents the most economical value, keeping in mind the loss of zinc in the lead concentrates, the loss of lead in the zinc concentrates and keeping the recoverable values as low as possible in the cleaner-jig tailings. Too often, in an endeavor to produce too high a grade of concentrates, the most economical procedure in operating this jig is completely overlooked.

Performance of the cleaner jig is described on page 907. Normally from 40 to 50 per cent of the total production of zinc concentrate is made on the cleaner jig and 70 to 75 per cent of the total production of lead concentrate. The grade of this zinc concentrate usually assays from 57 to 62 per cent zinc, and from 1 to 1.50 per cent lead. The grade of the lead concentrate usually assays from 79 to 84 per cent lead, and from 1.50 to 2.50 per cent zinc.

Chat-middling Circuit

The middlings or chats from the rougher jigs and the cleaner-jig tailings make up the middlings for re-treatment resulting from the primary concentration of the ore in the jigging department. The proper procedure at this stage in the re-treatment of these two middling products represents the major ore-dressing problem involved in the treatment of these ores. Many different circuits, therefore, are to be noted and much divergence of opinion exists. A few of the many types of middling circuits will be described:

1. The rougher chats are ground to minus 2 mm. by the so-called chat rolls in closed circuit with the so-called chat screen. The cleaner-jig tailings join the sands from the sand tanks, which collect the overflows from all of the desliming devices in the jig department, and are screened

over the so-called sand screen, usually with 1½ or 2-mm. openings. The oversize from the sand screen joins the rougher-jig chats for regrind in the chat rolls. The chat-screen product is put back over the rougher jig.

2. The rougher-jig chats and the cleaner-jig tailings are combined and rejigged, without grinding, over a secondary so-called chat roughing jig, where finished tailings are made and a middling product for re-treatment. This middling product is then ground to minus 2 mm. by means of rolls, rod mills or ball mills operating in closed circuit with the sand screen, the product of the sand screen going to the tables.

3. The rougher-jig chats and the cleaner-jig tailings are combined and ground to minus ⅛ in. by chat rolls operating in closed circuit with the chat screen, and the product is deslimed and jigged over the chat-roughing jig. This chat-roughing jig has been referred to in recent literature as a "sloughing jig." This jig produces a rich middling from the first two hutches which goes to the cleaner jig, a middling product and a tailing for discard. The middling product goes to rolls, rod mills or ball mills operating in closed circuit with the sand screen with 1½ or 2-mm. openings. The sand-screen product goes to the tables.

4. A modification of circuit 3 is to screen the cleaner-jig tailings over a 2-mm. screen and take the throughs direct to the tables, the oversize only going to the chat rolls for regrind.

5. Another modification of circuit 3 is to combine the middlings from the chat-roughing jig with the sands from the desliming devices in the jig department and grind this combined product to about minus 35 mesh in ball mills operating in closed circuit with mechanical classifiers, thus eliminating the tables and grinding all to flotation size.

6. Still another modification of circuit 3 is to grind just the middlings from the chat-roughing jig plus the oversize from a 2-mm. sand screen ahead of the tables to about minus 35 mesh in ball mills for flotation feed, the throughs of the sand screen going to the tables.

7. The rougher-jig chats plus the cleaner-jig tailings are ground to minus 2 mm. in the chat rolls in closed circuit with the chat screen. The product from the latter screen is deslimed and rejigged over a so-called "sand-cleaner jig," which produces finished concentrates and a tailing that generally is reground for table feed but in some cases is discarded.

8. No middling or chat rolls are used. All rougher-jig chats and cleaner-jig tailings are combined and sent to a rod mill operating in closed circuit with a 1.5-mm. sand screen. The throughs of the latter screen go to the tables.

9. A modification of circuit 8 is to grind to flotation size, thus eliminating the tables.

Many more modifications of these circuits exist, but the above serve sufficiently to illustrate the divergence of practice and opinion in regard to the proper procedure.

The following circuit, which is incorporated in only one of the newer mills of the district, differs materially from any of the circuits described above, and indicates an advance in the ore-dressing practice involved in connection with this step in milling, and for this reason will be discussed more in detail.

10. A part of the rougher-jig middlings or chats, consisting only of the lean disseminated chats or middling grains derived from the last two cells of the primary rougher jig, is ground to minus 2 mm. by chat rolls operating in closed circuit with the chat screen. The throughs of the chat screen are deslimed by means of an Esperanza drag-type classifier, the sand of which goes to a hydraulic classifying cone and the overflow to the slime thickener. The underflow of the hydraulic classifier is distributed to roughing tables, which produce finished concentrates and middlings for regrind in a ball mill operating in closed circuit with this hydraulic classifier and the roughing tables. The overflow from the hydraulic cone is minus 28-mesh material and joins the overflow from the desliming devices in the jigging department, which are collected in a large "live-feed drag" of the Esperanza type.

The cleaner-jig tailings, plus the rich chats or middling grains from the fourth cell of the primary rougher jig, are sent to the live-feed drag. The sands from the live-feed drag are ground to minus 2 mm. in sand rolls operating in closed circuit with the sand screen. The product of the sand screen is deslimed and makes up the live feed to the finishing tables; i.e., the tables that make tailings for disposal. The middlings from the finishing tables join the middlings from the roughing tables for ball-mill regrind.

This circuit has proved very effective in increasing the tonnage both of tailings eliminated and of concentrates produced from the table department, and by so doing has decreased the duty on the flotation department. It has also permitted the production of table tailings of a very satisfactory tenor, too low in value to justify further grinding for re-treatment by flotation.

Analysis of Middlings.—A physical analysis of the two middlings products to be dealt with discloses:

1. The lean disseminated chats or middling grains derived from the last cells of the rougher jig liberate some 95 per cent of their values when ground to minus 28 mesh in a ball mill, but a 2-mm. roll grind merely produces a larger number of smaller chats of the same specific gravity, too light to be concentrated into the middlings of the concentrating tables for regrind in the ball-mill circuit. This chat ranges in specific gravity from 2.7 up to 2.8. The specific gravity of the chert gangue is about 2.60.

2. The richer chats from the rougher jig, ranging in specific gravity from 2.8 up to that of blende, liberate a large percentage of their values when ground by rolls to 2 mm., and the resultant chats or middling grains not freed by a 2-mm. roll grind are of sufficient gravity to be readily

concentrated into the middlings of the finishing tables, thus permitting them to be reground in the ball-mill circuit. An appreciable percentage of barren chert also results from the 2-mm. roll grind, which can readily be discarded on the tables without further grinding.

3. The cleaner-jig tailings consist primarily of minus 2-mm. barren chert sand, plus a small percentage of rich chats. The 2-mm. sand makes very clean table tails without further reduction. The rich chats liberate some mineral upon regrinding through 2 mm., the resultant chats or middling grains being of sufficient gravity to be reclaimed for further grinding in the middling cuts on the finishing tables.

This physical analysis formed the premises for the last described circuit. It permits of the elimination of a large percentage of the barren chert sand on the finishing tables without being contaminated with lean chats of too low gravity to be concentrated into the table middlings. It takes advantage of the isolation and concentration of the lean chats brought about by the primary rougher jig without diluting this product with a considerable proportion of barren chert sand derived from the cleaner-jig tailing before subjecting it to regrind. It thereby keeps the tonnage of middlings requiring a fine grind for the liberation of their values very low. No chats or middling grains are combined with the table feed for the finishing tables until they are either freed of their values or have sufficient gravity to be concentrated into the middling cuts for regrinding in the ball-mill circuit.

Chat-roughing Jigs

The "chat-roughing jig" has been referred to in recent literature as the "chat-sloughing jig," but the terms are synonymous, both having for their object the elimination of barren chert gangue drawn by the chatters on the primary rougher jigs and that contained in the hutch products from the last two or three cells of the primary rougher jig.

The feed to this jig is derived as described on page 880. Usually it represents from 10 to 15 per cent of the mill-feed tonnage, this tonnage being dependent in large degree upon the practice followed in chatting the rougher jig, some operators believing that by drawing a large volume of rougher-jig chats the rougher-jig tailings can be kept to a minimum. Such practice, therefore, requires that the rougher-jig chat or middlings be reconcentrated for the elimination of the large volume of barren chert drawn by the chatters on the rougher jig. To this end this product is usually combined with the cleaner-jig tailing and the combined product ground to minus 1/8 or 3/16 in. in the chat rolls operating in closed circuit with the chat screen. The throughs of the chat screen are then deslimed either in automatic cones or drag classifiers of the Esperanza type. The sands from the desliming devices make up the feed to the chat-roughing jig. The roll grind usually liberates sufficient free mineral from the rich chats to produce a hutch product from the first two cells of a tenor suitable for cleaner-jig feed. The hutch product from the remaining cells, together with the bed draw, or so-called chat draw, represents a middling for re-treatment which is handled in a number of different circuits, as described under the preceding section "Chat-middling Circuit," and a tailing for discard.

The chat rougher-jig tailing eliminated will amount to 50 to 60 per cent of the feed to this jig, or some 5 per cent of the mill feed. The tenor of the middling produced usually is about twice the value of the feed to the jig. The tenor of the tailing produced is usually appreciably higher than the rougher-jig tailing, ranging from 1.50 to 2.00 per cent zinc. These tailings, after desliming usually in an automatic cone, are combined with the rougher-jig tailings.

In construction and operating details the chat-roughing jig does not differ materially from the other jigs. It usually consists of five cells, in series, the cell size varying from 36 by 42 in. up to 42 by 48 in. The fall between cells varies from 1½ to 3 in. The speed usually is about 160 to 180 r.p.m. The strokes vary from 1 to ¾ in. Some operators make a practice of putting on sieves small enough to permit the jig to bed itself. Others use coarser sieves and bed them with coarse chat bedding shoveled off the rougher jig. Usually the same type of chatter or bed draw is used as on the rougher jig.

This jig usually is attended to by the so-called "rougher man" who operates the rougher jigs.

The tailings produced, after being deslimed usually in an automatic cone, are combined with the rougher-jig tailings.

Tabling

All of the overflows and a portion of the sands (such as some of the rougher-jig smittem products, smittem products from the chat, sand or sloughing jigs, and the finer sizes of the cleaner-jig tailing) from the jigging department pass to a desliming device for separating sands for tabling and slimes for flotation. This device may be a large home-made belt drag (somewhat similar to the familiar Esperanza drag), three or four ordinary 9 or 10-ft. dia. 60° cones with spigot discharges, two or three large automatic cones (Boylan, Wood or DeMier, which function somewhat on the principle of the Allen cone), or a small "sand" thickener, 15 ft. in diameter with a heavily constructed raking mechanism. The rectangular sand tanks and V-tanks for this purpose are used rarely now, because they supplied the tables with a very irregular and nonuniform feed.

The overflow from the desliming device is sent to a thickener or thickeners (40 to 100-ft. dia.) serving the flotation department, and the sand discharge passes to the table-feed elevator, from which it goes to a 1½ to 2-mm. screen and thence to some type of classifying equipment. The screen oversize usually is tramp material and may be sent to a variety of places, such as to the chat jig or middling circuits, to a ball mill, to a rod mill, or to a set of rolls grinding this product alone.

A variety of screens is used at the head of the tables. The trommel, formerly the only kind used, has been displaced in many mills by some type of vibrating screen, such as the Leahy, the Tyler, the Robins, the Colorado Iron Works and some manufactured locally. Various shaking screens also appear occasionally for this duty. The trommel is inefficient, but its recommendations have been low first cost and high durability. Even the vibrating and shaking screens have not always been particularly efficient at 1½ mm., and some effort has been made to eliminate this screening duty by placing a coarse screen cloth (about 3⁄16-in. opening) on the screen and making it perform as a scalper for tramp material, and then relying on the first pocket of the classifying system to catch the plus 1½-mm. material and send it to whatever circuit is desired, either to a roughing table for production of some concentrate and a tailing or middling for grinding, or to a sloughing jig for making a tailing for waste and a middling product for grinding.

A great many kinds of classifiers have been used, some of them home-made and others manufactured. Ordinary 60° cones of various sizes and equipped with hydraulic water are in use. A combination of two such cones, one about 3 ft. in diameter to feed the "coarse-sand" tables and the other 4 to 5 ft. in diameter to serve the "fine-sand" tables, was popular a few years ago, but its use is on the wane. Classifiers of the Dorr, Akins, Butchart, the modified St. Joseph and home-made belt-drag types are in use in some of the plants. The prime object of all of the classifying equipment is the thorough removal of slime mineral below 150 mesh. Some operators practice classification to the extent of producing eight to ten products for as many tables; others maintain that the tableroom classifier has achieved its goal if it has accomplished thorough removal of minus 150-mesh material (not more than 3 per cent by weight) and they proceed to feed all of their tables with a product ranging from 1½ mm. (about 14 mesh) to plus 150-mesh mineral.

Some maintain that tables operate more efficiently with a thoroughly deslimed but unclassified feed and have introduced various odd-shaped riffles and riffles running at various angles on the table. Others, who favor classification, are inclined to a plain straight-line riffle, usually narrow, lying parallel to the line of motion of the table deck. Although theoretical considerations favor classification, no conclusive tests have been made in the district to prove the merits of either position. If the minus 150-mesh portion of the table feed is removed thoroughly, the difference in zinc tenor in the tailing from an unclassified and a classified feed would doubtless be small. One advantage of the classified feed separated into a number of products is that the middling grains are segregated in the coarse sizes on a few tables and can be sent in smaller quantities to the ball-mill circuit than if the middling grains are scattered over all of the tables, as from a deslimed but unclassified feed.

The number of tables employed in a mill is variable, but for a mill handling 45 to 50 tons of mine ore per hour, eight to nine tables are used; four to six are devoted to virgin feeds of sand, one to a zinc-chert middling, one to a lead-zinc middling, one occasionally as a zinc-concentrate table for recleaning and one for cleaning the flotation lead froth.

The overflow from the table classifiers passes to the slime thickener and their underflows to the sand tables, sometimes termed "coarse-sand" and "fine-sand" units. Usually the division between coarse sand and fine sand is about 48 mesh. Each sand table makes usually four products: a lead-zinc "cut," which is cleaned on a separate table; a finished zinc concentrate; a zinc-chert middling, which may be ball-milled or recirculated; and a tailing for discard. The tailing from the first one or two coarse-sand tables is sometimes a true middling product and may be retreated on a jig for further segregation of chat grains or may be ground; the tailing from the "fine" tables may be sent to flotation for further treatment if the desliming is poor.

Bucket elevators, sand pumps and diaphragm pumps are used for elevating products in the tabling operation; elevators are used extensively in many mills for heavy duty, but light loads are handled by sand-type centrifugal or diaphragm pumps.

The table zinc concentrate usually is mixed with the coarse jig concentrate and sold as gravity concentrate. Occasionally some of the concentrate from the "fine" tables is mixed with the flotation concentrate in the filter, but economically it is usually better to combine it with the jig concentrate, as often it commands a better market price than the flotation concentrate.

The tailings from the tables are pumped to waste, or dewatered and combined with the jigroom tailings on the tailing conveyor, or combined with all the other tailings from the mill in the tailing elevator.

The proportion of the total mill-concentrate production derived from the tables amounts to from 7 to 10 per cent. Usually the grade of the table concentrate is equal to or not more than 1 per cent lower than the jig concentrate. However, if there is considerable calcite in the mine ore, the grade of the table concentrate may be lowered considerably.

Usually the zinc is freed thoroughly from the chert at 35 mesh. Examination of the various sizes of a table tailing reveals that the coarser sizes that are above 35 mesh have a zinc tenor higher than the sizes from 35 to 150 mesh, which often may assay 0.33 to 0.5 per cent zinc. The minus 150-mesh size will assay 2.0 per cent zinc or more, but with excellent desliming of the table feed this size represents not more than

2 per cent of the weight of the total table tailing. The sizes above 35 mesh in the table tailing contain only a few tenths per cent of free mineral and the actual zinc tenor is dependent on the thoroughness with which chat grains have been removed or have been kept from getting into the table feed. Table tailings from the better plants assay from about 0.5 to 1.0 per cent zinc. This content depends primarily on how well the chats and the minus 150-mesh slime mineral have been removed.

The table department produces about 10 per cent of the total mill tailing. The tenor of the table feed usually is about that of the mill feed, whereas the tenor of the flotation feed ordinarily is twice or more that of the mine ore. The flotation department, although yielding usually twice as much concentrate as the table department, does not discard any more tailing. Plants that have abandoned tables entirely contend with twice as much tailing elimination in the flotation department and yet yield only 50 per cent more concentrate.

The district is divided in opinion as to whether to employ tables or eliminate them in favor of more fine grinding and more burden in the flotation circuit. *Those who advocate elimination of tables* cite as their principal arguments:

The tables are inefficient in separating chat grains, and hence yield high tailings.

The tables make a low-grade concentrate if calcite is present.

Tables make high tailings in the fines in some mills with indifferent desliming.

Flotation can produce a tailing of lower zinc content.

Economy of cost should result by operating one department instead of two.

Those who favor the use of tables state that:

Chats can be kept out of the table feed to a great degree or, if allowed to be in the feed, can be segregated by taking large cuts on the tables for regrinding or by use of sloughing jigs.

Low-grade tailings can be made if sufficient consideration is given to the chats and the slime.

Ball milling is expensive and its cost scarcely justifies the added mineral recovery.

Cost of producing a ton of tailing by flotation is more than the cost of producing the same amount by tabling.

Because of intermittent operation, interruptions are less serious as to mineral loss on tables than in flotation machines.

Initial cost of installation per ton of capacity is less for tables than for flotation equipment.

Those who favor use of tables contend it costs only about one-third as much to eliminate a ton of tailing by tables as by flotation, disregarding the cost of the regrind in ball mills required. In a mill well equipped with both tables and flotation the maximum difference in tailings is about 0.33 per cent zinc in favor of flotation, which is equivalent to $0.15 per ton of tailings on a $30 zinc concentrate market. It costs 12 to 15¢ more per ton of tailings to discard them by flotation than by tables, disregarding the cost of the ball-mill regrind necessary.

For the immediate future those opposed to tables will continue their efforts toward adjusting and adding to their fine-grinding and flotation equipment and seeking to strike a favorable balance between the added cost and the increased metallurgical recovery. Those favoring tables doubtless will continue their efforts to effect the best practice in desliming and in segregating the chats between 10 and 35 mesh, either by improved circuits in the table department or, preferably, by preventing as far as possible the presence of chat grains in the table feed.

Ball Milling

The original function of the ball mills first installed in the mills of this district was the regrinding of table middlings, which formerly were recirculated. By regrinding the table middlings it was found that the tenor of the table tailings could be reduced sufficiently to justify this regrind when the ore was "chatty." With free-milling ores, it was questionable whether such practice was warranted.

Following its introduction for merely regrinding table middlings, some operators found it advisable to further grind the coarse table tailings from, say, the first two pockets of a launder classifier, where the practice was to feed each table by a separate classifying pocket, in addition to the table middlings from all of the other tables. Such practice was predicated upon the fact that the table tailings from these coarse table sizes contained an appreciable percentage of lean chat grains too light to be concentrated into the middling cuts, but by grinding the entire tailing from about minus 2-mm. plus 28-mesh size to about minus 28 mesh, a large percentage of the values locked up in these lean chat grains were liberated, enough so to justify such a procedure.

Following upon this practice, advantage was taken of the concentration of the lean chats brought about by the rougher jig, such chat grains requiring fine grinding for the liberation of their values. Also, in addition to the table middlings, the middling products from the "chat-roughing jig" resulting from a re-treatment of the rougher-jig chats, were sent to the ball mill for regrind. By grinding such middlings to the point of unlocking before combining with the other table sands, it was possible to produce very satisfactory table tailings from the coarse table sizes as well as from the fine sizes.

Others went a step farther and proceeded to grind to flotation size all table sands and middling products resulting from the primary concentration of the ore in the jigging department, thus eliminating tables.

This procedure is predicated upon the contention that an economic table tailing could not be produced, but there is much difference of opinion as to this contention.

There are a number of different ball-mill circuits, a few of which are described below. Circuits 1 and 2 are most commonly used.

1. Grinding in open circuit with the tables, the table middling after dewatering being fed to the ball mill, the discharge of the latter returning to the table circuit.
2. Grinding in open circuit with the tables, the table middlings plus the table tailings from the coarse sand tables after dewatering being fed to the ball mill, the ball-mill discharge returning to the table circuit.
3. Grinding in closed circuit with a hydraulic classifier, the ball-mill feed consisting of table middlings, and the table tails from the coarse sand tables, the overflow of the hydraulic classifier returning to the table circuit.
4. A modification of circuit 3 using a mechanical instead of a hydraulic classifier, the overflow going to flotation.
5. A modification of circuit 4 being to grind all sands and middlings to flotation size, thus eliminating the tables.

With chatty ores, there can be no question as to the gain to be derived from ball milling, but just what should make up the feed to the ball mills, as well as just how far the grind should be carried, is far from a settled issue, the whole question hinging upon the economics involved more than upon the metallurgy.

Flotation

The flotation department is now one of the most essential phases of milling in the Tri-State district. Its function in general is the recovery of slimes that are not recoverable by gravity concentration. However, in a few instances it has supplanted gravity concentration by tabling.

The percentage of the total concentrate production derived from flotation has gradually increased in the past few years, due in part to a recognition of the importance of thoroughly desliming both jig and table feeds and sending such slimes to the flotation department rather than attempting to recover them by gravity concentration and thereby insuring high gravity tailing losses. While in the past there was a feeling that in practicing thorough desliming of the table feed the tables were being robbed in order to make a showing in the flotation department, the fallacy of this has gradually been recognized and the practice now in general is to send the table slimes to the flotation department rather than attempt their recovery on concentrating tables.

Ball milling of table middling products has in the past few years accounted for an appreciable increase in the flotation production. More efficient flotation operation, particularly since 1924, has also accounted in a large degree for increased production from the flotation department. At present an average of about 40 per cent of the total zinc concentrate production, both from mine ore and tailings retreatment, is being derived from flotation.

Grain Size of Flotation Feed.—In general, in order to effect a thorough enough desliming of the table feed to make possible the production of satisfactory table tailings, it has been necessary to remove most of the 150-mesh mineral and send it to the flotation department. On such a basis of mineral classification, it has been found that the splitting point between the flotation feed and the feed to the fine-sand concentrating tables lies around 65 mesh for the gangue.

The screen analysis in Table 2 is of the feed to the fine-sand tables after the feed has been thoroughly deslimed of minus 150-mesh mineral.

TABLE 2.—*Screen Analysis of Fine-sand Table Feed*

Mesh	Per Cent Weight	Cumulative Per Cent Weight	
		Plus	Minus
35	5.50	5.50	94.50
48	58.40	63.90	36.10
65	24.45	88.35	11.65
100	8.00	96.35	3.65
150	2.25	98.60	1.40
200	1.25	99.85	0.15
−200	0.15	100.00	

The screen analysis of a flotation feed (Table 3) will serve for comparison with Table 2 to bring out the splitting point between gravity and flotation concentration so as to permit the most economical over-all recovery by each respective department. In the comparison of these two analyses it is obvious that the dividing point between the fine-sand table sizes and

TABLE 3.—*Screen Analysis of Flotation Feed*

Mesh	Per Cent Weight	Cumulative Per Cent Weight	
		Plus	Minus
35	None	None	100.00
48	3.20	3.20	96.80
65	7.60	10.80	89.20
100	16.00	26.80	73.20
150	53.00	79.80	20.20
200	17.50	97.30	2.70
−200	2.70	100.00	

float sizes is above 65 mesh, as 88.35 per cent of the fine-sand concentrating table feed is plus 65 mesh and 89.20 per cent of the flotation feed is minus 65 mesh.

Grain Size of Flotation Concentrate.—From Table 4, it is obvious that only 9.35 per cent of the table concentrate is minus 150 mesh, whereas only 25.40 per cent of the flotation concentrate is plus 150 mesh. In other words, about 150 mesh is the splitting point between table and flotation concentrates when measured in terms of the grain size of the zinc mineral.

TABLE 4.—*Screen Analysis of Fine-sand Table Concentrates and of Flotation Concentrates*

Mesh	Per Cent Weight	Cumulative Per Cent Weight	
		Plus	Minus
	FINE-SAND TABLE CONCENTRATES		
35		None	None
48		None	100.00
65	16.35	16.35	83.65
100	52.95	69.30	30.70
150	21.35	90.65	9.35
200	8.95	99.60	0.40
−200	0.40	100.00	
	FLOTATION CONCENTRATES		
65	0.50	0.50	99.50
100	5.70	6.20	93.80
150	19.20	25.40	74.60
200	52.30	77.70	22.30
−200	22.30	100.00	

Tenor of Tailings Produced.—When the feed to the fine-sand tables has been deslimed of practically all of the minus 150-mesh zinc-mineral slime, it is possible to make a table tailing assaying some 0.30 to 0.40 per cent zinc; which, of course, is too low to permit further regrinding and flotation for any additional mineral that might be effected by flotation.

Under the degree of classification generally employed in table departments, satisfactory table tailing cannot usually be obtained unless the feed to the tables is deslimed of the minus 150-mesh zinc mineral and the flotation department therefore is called upon to effect the recovery of the minus 150-mesh mineral together with the finer slimes. Table 5 shows a screen analysis of the flotation tailing produced from a flotation feed as represented by Table 3.

The general tenor of flotation tailing losses ranges from 0.30 to 0.75 per cent zinc, depending in large degree upon the percentage of nonsulfide zinc mineral present and whether the ores are "free milling" or "chatty." Generally the economical point of unlocking lies somewhere between 28 and 35 mesh, although with chatty ores an appreciable percentage of the flotation tailing losses is due to locked mineral grains, which would require 80 to 100-mesh grind to liberate the zinc.

TABLE 5.—*Screen Analysis of Flotation Tailing from Feed Represented by Table* 3

Mesh	Per Cent Weight	Zinc, Per Cent	Zinc Distribution, Per Cent
Plus 35	None	None	None
48	4.60	0.64	3.85
65	11.50	0.86	12.95
100	16.10	0.66	13.82
150	45.40	0.76	45.16
200	19.40	0.70	17.74
−200	3.00	1.65	6.48
Composite		0.77	

Further analysis of the flotation tailing loss disclosed: Total zinc, 0.70 per cent; zinc as sulfide, 0.43 per cent; zinc as nonsulfide, 0.27 per cent.

Flotation Circuits.—Four different kinds of flotation circuits are employed, namely: (1) bulk flotation of zinc preceded by demudding, (2) bulk flotation of zinc, (3) differential flotation of iron and zinc and (4) differential flotation of lead and zinc.

1. When the flotation feed does not contain enough lead to justify a lead float, a flotation machine is sometimes used to pull mud or the so-called "colloids" before making the zinc float. This practice is generally being discontinued, because it has been found that under the proper reagent combination and control satisfactory grades of zinc concentrates can be produced even without cleaning of the rougher concentrate and without the use of the so-called "mud" flotation machine.

2. When sufficient lead is not present to justify a lead float, the usual practice now is to make a bulk flotation zinc concentrate and clean the rougher zinc concentrate. Such practice usually produces a rougher zinc concentrate assaying about 57.0 per cent zinc which after cleaning usually results in a grade of from 60 to 62 per cent. In some instances recleaning of this concentrate has been practiced, resulting in grades as high as 65.50 per cent zinc and commonly 64.50 per cent zinc, but such practice is to be condemned because of added cost of production and loss of concentrate from storage.

3. In some instances, where the iron content is high and there is only a trace of lead in the flotation feed, the iron has been successfully eliminated by floating it ahead of the zinc and discarding the iron con-

centrate so produced. Such practice has been very satisfactory in handling "irony" slimes.

4. Differential flotation of lead and zinc is the usual circuit employed and for this reason will be discussed in detail.

The flotation feed is derived from: (1) primary slimes produced from mining operations, (2) slimes produced from crushing and grinding in preparing the ores for jigging, (3) slimes produced from regrinding the jig middling products for re-treatment in the jig department, (4) slimes produced in preparing the sands for table concentration and (5) slimes produced from ball milling table middlings. Thus the percentage of the total mill feed going to the flotation department varies appreciably not only in the different mills, but from hour to hour in the same mill.

The slimes from these different sources are gathered together in the so-called slime thickener. The total tonnage of slimes varies from 10 to 20 per cent of the mill-feed tonnage and the total volume of water going to the slime thickeners varies from 2000 to 4000 gal. per min. In the usual plant, handling, say, 50 tons of mill feed, the flotation-slime thickener will be called upon usually to handle 7 tons of solids per hour and 3000 gal. of water. For this purpose a thickener 60 ft. in diameter is generally employed, and has proved satisfactory in most cases, except during extremely cold weather, when a small amount of lime, from 100 to 200 lb. per 10-hr. mill shift, is needed to clarify the thickener overflow. The overflow from the slime thickener generally goes direct to the mill pond or to a slime pond, where it is further clarified before returning to the mill heading water.

The feed to flotation is generally thickened up to about 25 per cent solids and pumped by means of a 4-in. diaphragm pump to either an elevator or sand-type centrifugal pump and elevated to the lead-flotation machine. Reagents generally are added just ahead of this machine. The usual practice is to float a low-grade lead froth assaying from 30 to 40 per cent lead and pump this entire froth to a concentrating table, where it is graded up to about 80 per cent lead. A lead middling is sometimes cut from this cleaner table and returned to the head of the flotation circuit. The backwater from this table, containing lead and zinc slime, is generally discarded, causing at times an appreciable loss of both lead and zinc.

Sometimes a rough lead concentrate is made, grading about 50 to 60 per cent lead, by circulating the lead froth from the last cells of the flotation machine to the head of the circuit, and taking this rougher froth to a lead cleaner machine where it is graded up to about 70 per cent lead, the cleaner tailings going back to the head of the rougher circuit. The cleaner lead froth either is pumped direct to the filter without prethickening, or goes to a small concentrate thickener where it is thickened to about 50 to 60 per cent solids before filtering. In the former case the froth usually is 30 to 40 per cent solids, and when ample filtering capacity has been provided gives no trouble; but sometimes when direct filtering is practiced, ample filtering capacity has not been provided and the filter cannot be kept from overflowing. When that occurs, the overflow is either returned to the head of the lead-flotation circuit or stored in a settling tank or small thickener and gradually fed back to the filter when the latter is not overflowing.

Generally the tailing from the lead machine passes direct by gravity into the first zinc-rougher machine, the zinc reagents being added to the pulp in the tail box of the lead machine. This does not allow for any appreciable time of contact for the reagents. Sometimes the reagents are added to the lead-machine tailings and then laundered to an elevator or pump and thence elevated to the zinc machine, thus allowing a few minutes of contact for the reagents before floating. Whether the latter offers any material benefit is open to question, as very little conditioning, if any, is required. The results from both circuits are about the same. It is the general practice to add all of the copper sulfate at the head of the zinc circuit and to stage the collecting and frothing reagents at a number of points in the circuit, but some plants have only one point of reagent addition, at the head of the circuit. Which practice is the best is also an open question, as both procedures produce satisfactory results.

The general practice now is to take off about a 57 per cent rougher zinc concentrate from the first few cells of the zinc machine and return the remainder of the froth to the head of the circuit. The rougher froth is then fed to a zinc cleaner machine either by gravity or by means of an elevator or pump. The cleaner machine produces a concentrate of from 60 to 62 per cent zinc when practicing single cleaning. When recleaning, 64 to 65 per cent grades are commonly produced. The cleaner tailings are returned to the head of the zinc roughers. In some plants, a zinc middling from the last cells of the zinc cleaner machine is returned to the head of the machine.

The zinc cleaner froth is generally pumped or elevated direct to the filters without prethickening. Usually it contains from 40 to 50 per cent solids, and on such a basis it is questionable whether prethickening is justified. In some few instances, though, zinc-concentrate thickeners are employed and the finished zinc froth is thickened before filtering. When direct filtering is employed provision is usually made to store the thickener overflow as it occurs, which is not often, or to return it to the head of the zinc-flotation circuit. In a few plants small concentrate thickeners have been installed to take the filter overflow when it occurs, from which, after thickening, it is pumped back to the filter. This practice makes direct filtering more desirable than prethickening when the percentage of solids in the froth from the cleaner machine can be maintained at from 40 to 50 per cent solids.

The concentrates from the filter generally discharge into small concentrate cars holding from 1000 to 1500 lb. wet weight, and are hand-trammed to the flotation-concentrate bins. Occasionally the concentrates discharge into small conveyors and are conveyed to the bins. The latter practice is preferable in the larger plants but the former suffices for the smaller plants.

The differential flotation of lead and zinc in this district is of particular interest, as very satisfactory results are being obtained without the use of any of the so-called addition or controlling reagents; i.e., reagents added either for the purpose of accelerating the flotation of lead or retarding the flotation of zinc. This is made possible no doubt by the amount of zinc sulfate present in the mine waters. The examples in Table 6 are typical of differential result being obtained without the use of cleaner machines for either the lead or zinc concentrates. Example 1 is representative of a high-lead feed and example 2 of a low-lead feed.

TABLE 6.—*Examples of Results without Cleaner Machines*

Products	Assays, Per Cent				Distribution, Per Cent			
	Zinc		Lead		Zinc		Lead	
	1	2	1	2	1	2	1	2
Flotation feed....	11.80	10.60	4.20	1.16	100.00	100.00	100.00	100.00
Lead concentrate	2.80	3.50	68.60	61.60	1.30	0.60	91.60	81.60
Zinc concentrate.	58.70	58.30	1.50	1.05	93.20	93.30	6.50	15.00
Tailing..........	0.86[a]	0.80[b]	0.10	0.05	5.50	6.10	1.90	3.40

[a] Zinc tailing loss showed only 62 per cent of the zinc present as sulfide zinc.

[b] Zinc tailing loss showed only 60 per cent of the zinc present as sulfide zinc.

The following reagent combination is typical of those used in effecting the above results:

Lead Circuit	Lb. per Ton Flotation Feed	Zinc Circuit	Lb. per Ton Flotation Feed
Yarmor pine oil..........	0.08	Copper sulfate............	1.00
Potassium ethyl xanthate..	0.10	Potassium xanthate.......	0.25
		Yarmor pine oil...........	0.02

It has been found that by practicing cleaning and recleaning of the lead rougher froth, lead concentrates ranging from 70 to 80 per cent can readily be made without incurring any appreciable increase in the lead losses and, of course, somewhat lowering the loss of zinc in the flotation lead concentrates. By cleaning and recleaning the rougher zinc concentrates, concentrates ranging in grades from 62 to 65 per cent can readily be produced without incurring any appreciable zinc losses.

Contaminants.—In a few instances tar occurs in the ores mined. Sometimes there is enough to necessitate the use of kerosene for cutting the tar from the lead and zinc minerals in order to make possible gravity concentration, and the kerosene acts as a contaminant in the flotation circuit. Sometimes there is not enough tar to cause any particular trouble in the gravity concentration, therefore kerosene is not used, but in such instances the tar itself acts as a contaminant in the flotation circuit.

Occasionally an appreciable amount of shale is present in the ores as mined and is sufficient to cause slime interference in the flotation circuits.

When these contaminants are present, it is difficult to make satisfactory flotation concentrate grades and tailings.

Beneficial Soluble Salts.—Most of the mine waters contain appreciable amounts of zinc sulfate in solution. It seems plausible that in most cases enough is present to be beneficial in effecting the differential flotation of lead and zinc, at least it has not been found necessary to add any reagents to effect a very satisfactory separation of the lead and zinc by flotation. The water analysis in Table 7 is of the mine water used in the plant where the flotation results just described were obtained.

TABLE 7.—*Analysis of Mine Water*

CONSTITUENT	PARTS PER MILLION	CONSTITUENT	PARTS PER MILLION
		Hydrogen (free acid)	None
Sodium	44	Carbonate (free alkali)	None
Calcium	329	Bicarbonate	293
Magnesium	79	Chlorine	7
Ferrous iron	2	Sulfate	1000
Ferric iron	18	Silica	7
Zinc	30		
Copper	0.0001	Total solids	1809

Deleterious Soluble Salts.—Some mills that derive their water supply partly or entirely from creeks fed by mine waters pumped from other mines and by surface drainage have trouble during seasons of heavy rainfall, due no doubt to organic compounds derived from the surface runoff or drainage and not from the mine waters. In some plants the use of lime has been effective during rainy seasons, but in others it has not proved beneficial.

An appreciable amount of ferrous salt in mine waters is detrimental to flotation, necessitating rectification before it can be used in mill heading water. A typical analysis of such a water is given in Table 8.

Reagents.—The reagents now generally used consist principally of cresylic acid or pine oil for frothers and potassium ethyl xanthate, sodium Aerofloat or Aerofloat No. 15 and No. 25 for collectors, Barrett No. 634 as a froth conditioner, copper sulfate as the zinc activator and occasionally a little sodium silicate as a dispersing reagent. At times lime is beneficial as a rectifying reagent.

TABLE 8.—*Analysis of Water Containing Deleterious Salts*

CONSTITUENT	PARTS PER MILLION	CONSTITUENT	PARTS PER MILLION
Ferrous iron	4.5	Cl	8.6
Ferric iron	25.5	Total acidity as H_2SO_4 (due to free acid, $FeSO_4$, $Fe_2(SO_4)_3$ and $ZnSO_4$)	854.
Ca	578.		
Mg	258.		
Zn	545.	Total dissolved solids	5332.
CO_2 (free)	15.6	pH less than 3.0.	
SO_4	3424.		

In a number of plants reagents are fed by suitable feeders, which permit better reagent control and lower reagent consumption, but in general reagents are fed with drip cans.

Reagent costs vary on an average from about 10¢ to about 20¢ per ton of flotation feed; or a reagent cost of 90¢ to about $1.50 per ton of concentrate produced. Costs of $3 to $4 per ton of float concentrate produced, however, are not uncommon in plants not subject to technical supervision.

Power Requirements.—The power requirements for the flotation department range from 5 to 10 hp. per ton of flotation feed per hour of running, of which about 75 per cent is required for operation of flotation machines. An average of 6.5 hp. per ton of flotation feed per hour would be representative of what is considered good practice.

Types of Flotation Machines.—The horizontal rotor flotation machine has been most generally used in the past. The pneumatic machines have not met with favor. At present the vertically driven impellor flotation machine is meeting with general acceptance. Comparison of the different kinds of machines has usually been reduced to the matter of initial cost and the cost of maintenance; the controlling factor being in small plants the initial cost and in larger plants, the machine maintenance cost.

Operating Costs.—Operating costs in terms of the cost per ton of flotation feed are not well known because the actual tonnage going to flotation is so variable and usually indeterminate a figure that only an approximation can be given. It will range somewhere between 35¢ and 50¢ per ton, depending in large measure upon tonnage handled.

Summary.—The usual method of feeding the gravity concentrating department from the crusher discharge causes a wide variation in the tonnage going to flotation from hour to hour. Therefore frequent adjustment is required in reagent and water supply, which makes almost impossible the nicety of reagent and density control that is so necessary to obtain the best results. The recent practice of providing storage bins between the concentrator and crushing plant has done much to alleviate this variable condition. Proper technical control has done more toward improving the efficiency of this department than any other one factor.

Disposal of Tailings

The land is so flat that tailings must always be elevated for disposal, either by tailing elevators or belt conveyors. Until 1923 the tailings elevator was used in almost every plant to dispose of all tailings; usually one elevator served the tailings from jig, table and flotation departments; occasionally the table and flotation tailings were served by a separate elevator and stored separately. The tailings elevator has the disadvantage of losing much headroom, and if a considerable tonnage is to be stored a number of "dummy" elevators are required on the tailings pile, often three to four "dummies" being used. The maintenance and care of these dummies, because of their location, is always difficult and expensive, particularly in cold or windy weather.

With the coming of more intensive desliming of gravity tailings and the demand for a dewatered and usually a deslimed and desanded tailing for market purposes, the stage was set for the introduction of the belt conveyor for this duty. Now many mills have conveyors, in spite of the fact that they are more expensive in first cost than the tailings elevator and present several new problems.

Before gravity tailings can be loaded on a belt conveyor, they must be dewatered thoroughly. The most satisfactory practice is to dewater the jig tailings and the table tailings separately, and place the coarse jig tailings on the conveyor at a point ahead of the table tailings, so that the jig tailings act as a bed for the table tailings. If both are dewatered in the same device, the table sizes either overflow and cause a high circulating load in the tableroom, or impart so much mobility to the whole tailing mass that considerable spillage results, causing trouble in the operation of the conveyor.

Thorough dewatering of these tailings results in high recovery of slimes for mill treatment, and leaves the tailings in excellent condition for sale as ballast, concrete admixture, etc. If the tailings are loaded directly from the conveyor to railroad cars, very little fresh water is needed at the top of the conveyor, but if they must be flumed from the conveyor, fresh water amounting to from 150 to 300 gal. per min. must be pumped to the top of the conveyor.

At some mills the flotation tailings are pumped by sand pumps to the top of the conveyor for fluming the coarser tailings; this should not be done if the tailings are to be reclaimed for sale. Pumping flotation tailings to this height for this purpose is expensive and necessitates screening of tailings if they are sold later for ballast. Pumping of flotation tailings to their own separate storage pond is common practice.

Mixing flotation tailings with jig tailings makes impossible as steep a slope to the tailing pile, because the fine tailing causes the entire mass to flatten, i.e., to assume a lower angle of repose and spread over more territory.

Many variations exist in dewatering the tailings for the tailing conveyor: Drags may be employed for dewatering and discharge directly to the conveyor, or automatic cones may partly dewater and discharge to a short tailings elevator, which in turn discharges to an automatic cone and then to a dewatering screen, which discharges the tailings to the conveyor.

The method of tailings disposal is dependent on several factors, and each plant has to weigh these factors and make a choice. In general, the simplest and usually the cheapest is by the tailing elevators, but if tailings are to be sold, or to be stored in greatest quantity per acre, the conveyor is to be preferred.

Disposal of Concentrates

The common method of disposing of mill concentrates is by hand shoveling. The concentrates from cleaning jigs discharge into a long shallow box alongside the jig and at the mill-floor level. After the water drains off, the concentrates are shoveled by the "jack" shoveler into the "ore" car. Water also drains from the concentrates in the storage "ore" bins, so that the moisture content of the shipped concentrate is usually from 2 to 3 per cent, and rarely exceeds 4 per cent. The "ore" car is made of light sheet metal mounted on trucks, its top being about 42 in. above the mill floor. It is pushed on light rails by the "jack" shoveler over the storage "ore" bins and dumped. The ore cars have capacities varying from 1000 to 2000 lb. of zinc concentrate; 50 to 60 per cent more of lead concentrate.

The table concentrates are handled in a similar manner. The lead and the zinc concentrates are caught in separate compartments of a box placed under the concentrate end of each table. Excess water overflows these boxes and the concentrate is shoveled into the ore car.

The mill crew keeps a record of the number of cars of each concentrate product sent to the storage ore bins per shift. This constitutes the only measure of the weights of concentrates recovered until the shipments are weighed and the ore bin is emptied, at which times "averages" or "shortages" are determined and adjustments made in the records and in the weight assigned each ore car.

In some mills the jig zinc concentrates and the table zinc concentrates go to an "ore" elevator, which in turn discharges to a large 8 to 10 ft. dia. dewatering cone of the Callow type. The cone is set high enough to discharge the dewatered concentrates directly into the ore car. In some installations the dewatering cone is replaced by a dewatering drag of the home-made Esperanza type.

The lead concentrates, both jig and table, are handled almost always by shoveling; this, however, is not a large task, as the quantity of lead is rarely more than one-sixth to one-fifth that of the zinc.

In every mill, the flotation zinc concentrates are filtered on vacuum filters. The moisture content of the filtered concentrate is usually about 11 per cent. The filters are mounted high enough to discharge directly into the ore car. Occasionally this concentrate is discharged to a conveyor belt, which discharges directly into the storage ore bin.

The gravity concentrates and the flotation concentrates are never mixed. They are dewatered separately and stored separately. Although all flotation zinc is filtered, only in some instances is the flotation lead filtered. Often the flotation lead froth is tabled, which results in both dewatering and concentration. The lead concentrate is caught in a box and from there shoveled into the ore car. The very small tonnage of flotation lead usually does not justify ample recleaning equipment and filtering machinery, hence the somewhat makeshift method of handling this material.

The selling custom of the district always has been for the ore buyer to purchase the concentrate where it lies in the bins; therefore the producer has not developed facilities for further handling of the concentrates.

The ore bins for concentrates storage vary in size; usually they are 26 ft. wide and as long as the producer may need for storage requirements. All of these bins are built on the ground. Their bottoms are earth, wood or concrete; they may have sides or not; if they do, the sides are not higher than 42 in. from the ground level, so that the buyer can shovel easily from the bin into his truck. The ore track from the mill runs lengthwise over the bin. This track is at mill-floor level; about 6 to 13 ft. above ground level.

Ore bins usually are open to the elements. Wind and rain cause considerable losses; therefore some mills have roofs and sides for their bins, the sides having many doors, to permit easy removal of the contents.

Virtually all mills have truck scales, so that the producer weighs the buyer's truckload as it passes from the ore bin to the railroad car.

Water Supply and Requirements

The sources of the water supply are creeks, deep wells and mine water, the chief source being the latter. The pollution of the creek water by surface drainage sometimes makes it unfit for use as mill water in the flotation department. Some mine waters, also, are not suitable, on account of deleterious salts such as large amounts of ferrous sulfate. Deep-well water is used when a good supply of creek water or mine water is not available.

Water requirements vary from 3000 gal. per min. when milling about 50 tons per hour, up to 4000 gal. per min. when milling 100 tons per hour, giving a water ratio of about 15 to 1 in the first instance and about 10 to 1 in the latter. Of the entire amount of water required, about 65 per cent is required for the jig department, 25 per cent for the tables and 10 per cent for the flotation department. The general practice is to have a separate water supply for each department. In a mill handling 50 tons per hour an 8-in. enclosed, low-head centrifugal pump for the jig department, a 4-in. centrifugal for the table department and a 3-in. for the flotation department usually are adequate. Header lines usually run underneath the mill floor from each pump and risers are taken from these headers to each piece of equipment requiring water. The total head rarely exceeds 50 ft. Such installations give practically no trouble and no extra help is required to watch the pumps, as they are attended by the regular mill crew. Formerly these pumps were belt-driven from the main-line shaft in each department. The general practice now is to use motors directly connected to the pumps, thus permitting the pumps to be started before the rest of the mill equipment.

The flow sheets employed generally do not allow the water to be re-used in the mill, all such mill-heading water going to the slime thickener and the overflow from the latter returning to the mill ponds. The mill-supply water is impounded in mill ponds varying in area from a few hundred square feet up to several acres. The usual practice is to employ two mill ponds, the mill tailings and the slime thickener overflow going to one where the slimes are settled out, and the overflow from the tailing pond going to a clear-water pond for re-use as mill-heading water.

Practically all mill water is thus reclaimed for re-use. During the summer months evaporation and seepage generally cause losses that require from 50 to 100 gal. per min. of make-up water.

Power Supply and Requirements

Early operations in this district used steam power, as neither gas nor electric current was available. When natural gas was piped into the field, gas engines replaced most of the steam plants, although even today a few plants still use steam. When electric power became available, the gas engines were replaced by motors to a considerable extent. Electric power is generally employed in the larger plants, whereas the smaller operations use gas engines.

The choice between electric power and gas-engine power lies mainly in the load factor. In single-shift operations with periodic interruptions due to curtailment programs in vogue in the district, or a temporary suspension of operations due to low metal prices, much is to be said in favor of gas engines vs. electricity, as a considerably lower power cost can be obtained. With present gas rates power costs of from 0.90¢ to 1.25¢ per horsepower-hour are being obtained even under conditions of interrupted operations and low power factor. Under such conditions of operation the present electric power costs would be from 1.25¢ up to 1.50¢ per horsepower-hour; in some instances even higher.

The main advantage, therefore, in the use of gas engines lies in the fact that the power cost does not depend upon the load factor, whereas with electric power it is the important consideration if low power costs are to be obtained. Both sources of power are more than adequate for the demand and therefore are equally reliable. In plants operating 24 hr. per day and handling large tonnages, giving a high load factor, a power cost of about 1¢ per horsepower-hour can be obtained with electric current under the present power rates.

The two-cycle gas engine is generally used, showing a gas consumption of some 17 cu. ft. per horsepower-hour. Four-cycle engines, while showing lower gas consumption, around 12 cu. ft. per horsepower hour, are not popular because of higher repair and maintenance cost. Electric power is available delivered to the mines as 25-cycle, three-phase, 2300-volt current. The practice is to use 2300-volt motors down to 25 hp. and 220-volt motors under 25 hp. in the mill, and 220-volt motors for hoisting and mine service.

The Commerce Mining and Royalty Co. has its own central power plant, capable of generating 10,500 electric hp., employing diesel engines. It distributes this current to its own mines over its own transmission lines. The same company has a central compressor plant of sufficient capacity to supply all the company's mines through a system of air mains serving practically the area of the entire mining district. It is claimed that the economies effected by this central power and compressor plant have more than justified the initial cost of the plant.

The power required for milling ranges from 7.5 up to 10 hp. per ton milled. The usual practice is to drive all mill equipment from one main-line shaft in each department, employing separate motors to drive each department. Few reliable data are available for the power required for the individual pieces of equipment, but the following distribution approximates the actual requirements for the average mill handling 50 tons per hour: crushing and screening, 125 hp.; jigging, 35; regrinding (rolls), 50; tabling, 25; flotation, 50; ball milling, 75; water supply, 50; total, 410 horsepower.

The present practice is toward more individual motor drives, permitting greater flexibility in operation. In one mill practically all line shafts have been eliminated. While separate motoring necessitates higher first cost and lower over-all motor efficiency, the flexibility of operations has much in its favor.

METALLURGICAL RESULTS

Desliming of Rougher-jig Feed.—Table 9 gives performance details of a belt drag of the Esperanza type, employed as a desliming device preceding a rougher jig. The distribution of the products as well as the water ratio varies appreciably in the different mills. The water ratio is dependent upon the amount added in the crushing of the ores, which is governed in large degree by the varying fall of the launders in the different mills. The fineness of the ore or feed depends upon the size being crushed to and the method of mining.

TABLE 9.—*Performance of Desliming Drag*

PER CENT WEIGHT

Size, Mesh	Feed	Slimes	Sands
+8	55.64	0.00	63.17
−8 + 35	25.30	0.18	28.70
−35 + 65	5.55	2.40	5.98
−65 + 200	6.39	45.01	1.16
−200	7.12	52.41	0.99
Totals	100.00	100.00	100.00
Zinc, per cent	5.58	12.80	4.60
Zinc distribution, per cent	100.00	27.39	72.61
Tonnage distribution, per cent	100.00	11.92	88.08
Tons per hour	30.00	3.60	26.40
Water, gal. per min	530.00	510.00	20.00
Water ratio	4.41	35.63	0.19

Desliming efficiency @ −65 mesh, per cent	85.98
Dewatering efficiency, per cent	96.22

Performance of Rougher Jig.—Table 10 gives the performance of a rougher jig and gives an idea of the percentage of smittem, chats and tails produced as well as their assay values and metal distribution.

TABLE 10.—*Performance of Rougher Jig*

Product	Per Cent Weight	Zinc, Per Cent	Zinc Distribution, Per Cent
Feed	100.00	7.53	100.00
Smittem	19.47	32.27	83.46
Chats	3.01	11.75	4.70
Tailings	77.52	1.15	11.84

SCREEN ANALYSIS OF FEED

Screen Size, Mesh	Per Cent Weight	Zinc, Per Cent	Zinc Distribution, Per Cent
+6	58.50	7.32	56.90
−6 + 14	28.40	5.39	20.34
−14 + 28	7.20	10.86	10.39
−28 + 65	4.10	11.71	6.38
−65	1.80	25.06	5.99
Totals	100.00	7.53	100.00

SCREEN ANALYSIS OF TAILING

Screen Size, Mesh	Per Cent Weight	Zinc, Per Cent	Zinc Distribution, Per Cent
+6	57.44	1.26	63.12
−6 + 14	30.68	0.81	21.45
−14 + 28	6.77	0.68	4.01
−28 + 65	3.90	1.42	4.79
−65	1.21	6.30	6.63
Totals	100.00	1.15	100.00

Screen Analysis of Rougher-jig Feed and Tails.—Table 11 gives an idea of the distribution of values. The screen analyses in this table are typical of a "chatty" ore considerably higher in values than the usual mine run.

TABLE 11.—*Screen Analyses of Rougher-jig Feed and Tailing*

Screen Size	Per Cent Weight	Assays, Per Cent		Metal Distribution			
				Weight		Percentage	
		Zinc	Lead	Zinc	Lead	Zinc	Lead
				FEED			
Inch							
+⅜	17.82	5.10	0.55	0.909	0.098	11.97	5.92
−⅜ + ¼	24.48	6.70	1.20	1.640	0.294	21.59	17.76
−¼ + ⅛	26.40	6.50	1.25	1.716	0.330	22.59	19.94
Mesh							
−⅛ + 10	8.17	6.90	1.20	0.564	0.098	7.42	5.92
−10 + 20	8.68	8.50	2.53	0.738	0.219	9.72	13.23
−20 + 35	6.79	10.70	3.20	0.726	0.217	9.56	13.11
−35 + 48	2.11	13.40	3.55	0.283	0.075	3.73	4.53
−48 + 65	1.79	14.60	3.40	0.261	0.061	3.43	3.69
−65	3.76	20.20	7.00	0.759	0.263	9.99	15.90
Totals............	100.00			7.596	1.655	100.00	100.00
				TAILING			
Inch							
+⅜	17.04	0.66		0.112		12.17	
−⅜ + ¼	27.68	1.02		0.282		30.65	
−¼ + ⅛	32.48	1.04		0.338		36.74	
Mesh							
−⅛ + 10	10.35	0.96		0.099		10.76	
−10 + 20	8.60	0.64		0.055		5.98	
−20 + 35	3.00	0.46		0.014		1.52	
−35 + 48	0.35	0.58		0.002		0.22	
−48 + 65	0.16	0.90		0.001		0.11	
−65	0.34	5.04		0.017		1.85	
Totals............	100.00			0.920		100.00	

	Feed		Tailing	
	Zinc, Per Cent	Lead, Per Cent	Zinc, Per Cent	Lead, Per Cent
Head-sample assay............	7.500	1.450	0.92	0.10
Calculated head...............	7.596	1.655	0.92	N.D.
Difference....................	0.096	0.205	Nil	

Cleaner-jig Performance.—Table 12 shows performance of a seven-cell, 36 by 42-in. Cooley cleaner jig.

TABLE 12.—*Performance of Cooley Cleaner Jig*

Cell No.	1	2	3	4	5	6	7
Stroke, in.	1¾	1½	1¾	1¼	1¼	1	¾
Sieve, in.	3/16	3/16	¼	¼	3/16	⅛	⅛
Speed	164 r.p.m.; size 36 by 42 inches.						

Products	Weight, Tons per Hour	Per Cent Weight	Assays, Per Cent		Metal Distribution, Per Cent	
			Zinc	Lead	Zinc	Lead
Feed	7.60	100.00	32.00	13.80	100.00	100.00
Zn concentrate	3.82	50.21	58.60	1.60	91.95	5.82
Pb concentrate	1.22	16.04	2.70	80.20	1.35	93.20
Tailings	2.56	33.75	6.35	0.40	6.70	0.98

SCREEN ANALYSES OF LEAD AND ZINC CONCENTRATES

Screen Size	Weight Distribution, Per Cent	
	Zinc	Lead
Inch		
−⅜	0.0	0.0
Mesh		
−⅜ + 8	29.13	30.20
−8 + 20	34.15	32.20
−20 + 30	17.66	14.15
−30 + 60	13.72	9.20
−60	5.34	14.25
	100.00	100.00

Zinc concentrate assayed 2.07 per cent Fe and 0.89 per cent CaO; lead concentrate assayed 1.30 per cent Fe and 0.37 per cent CaO.

Sand-jig Cleaner Performance.—Table 13 gives the performance of a five-cell, 36 by 42-in. sand-jig cleaner, the feed consisting of the rougher jig chats plus the cleaner tails ground to minus 2½ mm. and deslimed in a dewatering box.

TABLE 13.—*Performance of Sand Cleaner*

Products	Weight Per Cent	Zinc, Per Cent	Zinc Distribution	
			Weight	Percentage
Heads	100.00	7.75	7.75	100.00
Zinc concentrate	9.71	57.00	5.54	71.44
Tailings	90.29	2.45	2.21	28.56

SCREEN ANALYSIS OF FEED

Screen Size	Per Cent Weight	Assay, Per Cent Zinc	Zinc Distribution	
			Weight	Percentage
Mesh				
+10	6.30	8.50	0.542	7.07
+20	41.60	7.35	3.057	39.89
+35	36.69	6.10	2.238	29.20
+48	6.86	7.30	0.501	6.54
+65	3.71	10.45	0.387	5.05
−65	4.84	19.40	0.939	12.25
Totals	100.00		7.664	100.00

SCREEN ANALYSIS OF TAILS

Mesh				
+10	11.04	1.84	0.203	9.31
+20	43.27	1.82	0.787	36.08
+35	31.15	1.48	0.461	21.14
+48	6.20	1.56	0.097	4.45
+65	3.61	2.42	0.087	3.99
−65	4.73	11.55	0.546	25.03
Totals	100.00		2.181	100.00

	ZINC, PER CENT
Head assay	7.75
Calculated head	7.66
Difference	0.09

Chat-roughing Tables.—In a very few instances the sand-jig cleaner tailings are tabled over a large-deck concentrating table for recovery of the chat, or middlings, into an enriched product and free mineral, which the sand jig failed to catch. The tests listed in Table 14 of this chat-roughing table show the performance of such a procedure.

TABLE 14.—*Tests on Chat-roughing Table*

Products	Per Cent Weight	Zinc, Per Cent	Zinc Distribution, Per Cent
Test No. 1			
Feed	100.00	3.40	100.00
Middlings	36.30	6.58	70.30
Tailings	63.70	1.59	29.27
Test No. 2			
Feed	100.00	3.47	100.00
Middlings	38.70	6.51	71.20
Tailings	61.30	1.63	28.80
Test No. 3			
Feed	100.00	3.46	100.00
Middlings	73.70	4.30	91.60
Tailings	26.30	1.10	8.40

Chat-roughing Jig.—The performance of a chat-roughing jig in Table 15 gives an idea of the percentage of tailings discarded and the enrichment of the chats effected.

TABLE 15.—*Performance of Chat-roughing Jig*

	Per Cent Weight	Zinc, Per Cent	Zinc Distribution	
			Weight	Percentage
Mill feed	100.00	6.70	6.700	100.00
Middlings	47.99	12.01	5.764	86.20
Tailings	52.01	1.80	0.936	13.80

Table Classification.—Table 16 indicates the tableroom classification.

TABLE 16.—*Table Classification*

Products	Screen Sizes, Mesh (Tyler)								
	+20	+28	+35	+48	+65	+100	+150	+200	−200
1. Sand-drag feed	1.80	6.70	8.80	37.00	18.50	10.90	6.50	6.40	3.40
2. Sand-drag overflow			0.15	2.20	8.00	12.60	44.70	22.55	9.80
3. Sand-drag sands	2.80	6.00	12.10	41.30	20.80	11.10	3.80	1.50	0.60
4. Overflow of coarse-sand cone			0.80	21.40	41.90	17.80	10.70	5.20	2.20
5. Coarse-sand table feed	4.30	13.70	18.10	44.60	12.40	4.50	1.50	0.60	0.30
6 Overflow of fine-sand cone				5.00	36.50	24.50	20.10	9.60	4.30
7. Fine-sand table feed			0.30	52.70	35.50	8.90	2.10	0.40	0.10
8. Overflow from V-tank				0.40	1.10	4.80	50.60	36.50	6.60
9. Fine table zinc concentrate				0.30	16.10	55.70	2 .00	4.20	0.70

In the circuit shown, product 1 is the feed to a desliming drag of the Esperanza type at the head of the table department, such feed being derived from a V-bottom sand tank, which collects all the sand and slime products produced in the jig department. The overflow of the sand tank, together with products 2 and 6 (the overflows from the sand drag and the fine-sand hydraulic classifying cone) make up the feed to the slime thickener ahead of the flotation department.

Product 2 is a slime product overflowing the sand drag, and goes to flotation. Product 3 is a deslimed sand product, or the feed to the first hydraulic classifying cone, designated as the coarse-sand cone. Product 4, the overflow of the first hydraulic cone (the so-called coarse cone) makes up the feed to the second hydraulic cone, designated as the fine-sand cone. Product 5 is the underflow of the coarse-sand cone and makes up the feed to tables working on coarse sands. Product 6 is a slime product going to flotation. Product 7 is the underflow of the fine-sand classifying cone and makes up the feed for tables working on fine sands.

The tenor of the table tailings produced when grinding the table middlings in a ball mill is:

	PER CENT ZINC	
	MINIMUM	MAXIMUM
Coarse sand (−20 + 35 mesh)	0.50	0.70
Fine sand (−35 + 65 mesh)	0.30	0.50

Before table middlings were ground, when they were merely recirculated, the tenor of the table tailings was as follows:

	PER CENT ZINC	
	MINIMUM	MAXIMUM
Coarse sand (−20 + 35 mesh)	1.25	1.80
Fine sand (−35 + 65 mesh)	0.30	0.50

It should be noted that the fine-sand table concentrate contains only 4.90 per cent of minus 150-mesh mineral, and it has been found necessary to deslime table feeds of the minus 150-mesh mineral, in order to produce table tailings with the limits of assay given. Failure to recognize this factor has accounted in large degree for high table tailings, and in some cases, in conjunction with other factors, for tables being eliminated in favor of flotation.

Ball Milling.—The data in Table 17 indicate the economic advantages to be derived from ball milling chatty ores in which rolls do not suffice to liberate the values in the disseminated chats. In this particular case the decrease of 0.33 per cent zinc in the mill tailings was due entirely to the ball-mill regrind, practically all the increased production being accounted for in the flotation department, as shown by the data in the second section

TABLE 17.—*Effect on Ball Milling of Chatty Ores*

Period[a]	Mill Feed, Per Cent Zinc	Concentrate Recovery, Per Cent	Tailing, Per Cent Zinc
1	7.13	10.09	1.33
2	6.76	10.08	1.00

TABLE 17.—(*Continued*)

DISTRIBUTION OF MINERAL IN MILL FEED, PER CENT

Period	Feed Mill	Zinc Concentrate				Lead Concentrate		Tails
		Cleaner Jig	Sand Jig	Table	Flotation	Cleaner Jig	Flotation	
1	100.00	45.86	10.89	6.38	19.43	0.40	0.19	16.85
2	100.00	45.40	9.17	5.60	25.90	0.26	0.14	13.53

SCREEN ANALYSIS OF BALL-MILL CLASSIFIER OVERFLOW

Size, Mesh	Per Cent Weight	Zinc, Per Cent	Zinc Distribution, Per Cent
Degree of Grinding Required to Unlock Some 95 Per Cent of the Values			
+28	5.30	1.00	1.44
+35	11.00	1.18	3.55
+48	23.50	1.58	10.15
+65	17.00	2.50	11.63
−65	43.20	6.20	73.23
Composite..........	100.00	3.66	100.00
Degree of Grinding Required to Unlock Some 87 Per Cent of the Values			
+28	12.00	1.70	5.55
+35	14.70	1.78	7.10
+48	21.80	1.96	11.65
+65	13.50	2.76	10.15
−65	38.00	6.34	65.55
Composite..........	100.00	3.68	100.00

[a] Period 1 was before the installation of the ball mill, and period 2 was following its installation.

of Table 17. When grinding to 5.3 per cent on 28 mesh, the mill handled 5 tons per hour. When grinding to 12.00 per cent on 28 mesh, 6 tons per hour could be handled. The latter grind was adopted as representing the most economical procedure when all other contributing factors were taken into account, such as the return for further grinding of the rich chat grains in the plus 28-mesh size from the subsequent middling cuts from the tables. In other words, the latter grind produced as low coarse table tailings as the finer grind.

A summary of the total benefits derived from ball milling in this particular case showed:

1. The final mill tail was reduced from 1.33 per cent to 1.00 per cent zinc.

2. The table zinc concentrate was raised in grade from 57.08 per cent to 59.11 per cent zinc.

TABLE 18.—*Composite Metallurgical Results on Chatty Ore*

Products	Per Cent Weight	Assays, Per Cent		Distribution, Per Cent	
		Zinc	Lead	Zinc	Lead
Mill feed	100.00	8.07	0.96	100.00	100.00
Zinc concentrate					
Cleaner Jig	6.55	59.00	0.83	47.80	5.60
Sand Jig	1.00	57.00	1.10	7.10	1.10
Table	1.14	59.00	0.43	8.30	0.50
Flotation	3.45	57.00	1.33	24.40	4.70
Lead concentrate					
Cleaner Jig	0.85	1.00	85.00	0.10	75.60
Table	0.06	1.60	80.10		5.00
Flotation	0.09	2.75	59.70		5.70
Composite tailings	86.86	1.14	0.02	12.30	1.80

	PER CENT
Total zinc-concentrate recovery	12.14
Total lead-concentrate recovery	1.00
Total concentrate recovery	13.14
Total zinc in zinc concentrate	87.60
Total lead in lead concentrate	86.30

TABLE 19.—*Composite Metallurgical Results on Free-milling Ore*

Products	Per Cent Weight	Assays, Per Cent		Distribution, Per Cent	
		Zinc	Lead	Zinc	Lead
Mill feed	100.00	2.72	1.28	100.00	100.00
Zinc concentrate					
Cleaner jig	1.90	61.00	1.18	42.60	1.70
Table	0.55	60.60	0.13	12.10	0.50
Flotation	1.01	57.80	1.43	21.30	1.10
Lead concentrate					
Cleaner jig	1.08	0.85	84.10	0.40	70.80
Table	0.06	0.80	83.70		3.90
Flotation	0.36	3.00	56.80	0.40	16.10
Composite tailings	95.04	0.67	0.08	23.20	5.90

	PER CENT
Total zinc-concentrate recovery	3.46
Total lead-concentrate recovery	1.50
Total zinc in zinc concentrate	76.00
Total lead in lead concentrate	90.80

3. The percentage of lead in the table concentrate was decreased from 1.15 to 0.52 per cent.

4. The coarse table tailings were reduced from 1.65 to 0.63 per cent zinc.

5. Losses incurred in discarding a tailing from a sand-roughing jig, previous to the ball-mill installation, were eliminated.

Flotation Metallurgical Results.—See the section on page 890 for a discussion of this phase of the milling.

Over-all Metallurgical Results Obtained.—Tables 18 and 19 illustrate about the average results that can be obtained on: (1) a chatty ore of high values and (2) a free-milling ore of low values; representing extremes.

Tailings Re-treatment.—Table 20 shows results obtained in re-treatment of tailings that locally would be classed as "chatty," which are those most refractory to re-treatment.

TABLE 20.—*Results of Re-treatment of Tailings*

Products	Weight, Tons	Per Cent Weight	Assays, Per Cent Zinc	Zinc Distribution	
				Weight	Percentage
Mill heads	209,663.00	100.00	2.391		100.00
Zinc concentrate					
Table	1,917.92	0.91	59.708	0.543	22.47
Flotation	3,136.21	1.50	61.534	0.923	38.20
Composite tailings	204,608.87	97.59	0.974	0.950	39.33
Calculated heads				2.416	

CONSTRUCTION COSTS

Mill-construction costs vary over a wide range, depending upon the amount of used machinery and equipment and building materials purchased or salvaged from other mills. Very seldom are all new machinery, equipment and building materials used, but in such cases mill costs run about $2000 per hourly tonnage of capacity; that is to say, a mill having a capacity of 50 tons per hour will cost around $100,000.

The following figures are representative of the percentage of the total cost of one of the larger mills for which all machinery, equipment and building materials were new:

	PERCENTAGE OF TOTAL COST
Building ore bins and other structures	21.11
Machinery and building foundations	8.42
Machinery and equipment	58.07
Installation of machinery and equipment	12.40
Total	100.00

OPERATING COSTS

The total cost of milling varies from a minimum of about 30¢ per ton, based upon a milling rate of 75 to 100 tons per hour, up to about 50¢ per ton, based upon a milling rate of 15 to 20 tons per hour. The average cost is about 35¢ per ton, based on the average milling rate of 40 to 50 tons per hour. Table 21 is indicative of the costs obtained.

TABLE 21.—*Operating Costs*

	Cost per Ton Milled		
	Maximum	Average	Minimum
Labor	$0.22	$0.11	$0.07
Supplies and repairs	0.14	0.12	0.14
Power	0.14	0.12	0.08
Total	$0.50	$0.35	$0.29

Labor.—The usual mill crew required for operating labor and the normal wage scale, based on a reasonable concentrate market and a 10-hr. day, are listed in Table 22. Until recently[1] the mills normally operated six 10-hr. shifts per week. So far as possible all mill repairs are made on Sunday, as many of the mill crew as necessary being used for such work. The crew listed in Table 22 handles up to 50 tons per hour. By the addition of two more men, one at $3.75 and the other $3.00 per day, it can handle as much as 100 tons per hour. When handling only 20 tons per hour one jack shoveler and one tailing herder can be dispensed with.

TABLE 22.—*Customary Mill Crew and Wage Scale*

Labor Classification	Rate, Per Day	Weekly Payroll
One mill foreman		$ 40.00
One crusher feeder	$3.25	19.50
One rougher-man	3.75	22.50
One cleaner-man	3.75	22.50
One table-man	3.75	22.50
One float-man	4.00	24.00
Two jack shovelers	3.00	36.00
One tailing herder	3.00	18.00
		$205.00

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[1] At present most operations are on an 8-hr. shift basis, and mills may operate one' two or three shifts per day. Tailings re-treatment mills usually operate continuously, either using three 8-hr. or four 6-hr. shifts. Wages have been readjusted accordingly, to a 40 to 42-hr. week.

For milling 20 tons per hour or 1200 tons per week, the operating labor costs *per ton milled* would be $0.141; for milling 50 tons per hour (3000 tons per week) they would be $0.068 and for 100 tons per hour (6000 tons per week) they would be $0.041.

The difference between these operating labor costs and the total labor costs as given under cost of milling is represented by repair labor, the figures above indicating the decrease in operating labor resulting from increased tonnages milled.

Supplies and Repairs.—Very few of the companies attempt to split up this account into its component parts, merely charging all milling costs, exclusive of labor and power to "Supplies and Repairs." In most cases no attempt is made ever to split up the total power costs into those required for milling and mining.

The analysis of this "Supplies and Repairs" account in Table 23 covers one years operation.

TABLE 23.—*Cost of Supplies and Repairs*

ANALYSIS OF ACCOUNTS FOR ONE YEAR

	COST PER TON MILLED		COST PER TON MILLED
Maintenance and Repairs		Maintenance and Repairs	
Crusher	$0.0013	Building and ore bins	0.0017
Rolls	0.0399	Laundering and spouting	0.0067
Screens	0.0060	Mill lighting	0.0012
Elevators	0.0023	Fire-protection equipment	0.0004
Jigs	0.0037	Balls for grinding	0.0063
Tables	0.0086	Flotation reagents	0.0273
Flotation machines	0.0002	Lubrication	0.0059
Filter	0.0016	Heating	0.0025
Pumps	0.0008	Tools	0.0007
Drag classifiers	0.0002	Ice and drinking water	0.0007
Hydraulic classifiers	0.0010	Testing (assaying)	0.0005
Automatic cones	0.0004	Miscellaneous	0.0011
Transmission	0.0068		
Piping	0.0024	Total cost of supplies and repairs	$0.1302

Power.—Very few of the operators keep separate cost figures on mill power, and for this reason few data are available in this connection. It is safe to say, however, that some 7.5 to 10 hp. is required per ton milled, the amount being dependent upon the tonnage milled and the physical arrangement of mill equipment.

When using electric power on the basis of single-shift operation, 10 hr. per day, current costs vary from $0.0136 to $0.0150 per kilowatt-hour. On the basis of 24 hr. operations a current cost of about $0.0110 per kilowatt-hour can usually be obtained, owing to the increased current consumption for a given connected load. In other words, there is about 19 per cent saving in power in 24-hr. operation over single-shift operation, which is an item worthy of more consideration than it has received when a reduction in operating costs is imperative. Table 24 gives an idea of power requirements and cost.

Table 24.—*Power Requirements and Their Cost at One Mill*

Connected load:	
Crushing plant	3.490 hp. per ton crushed
Concentrator	5.125 hp. per ton milled
Total connected horsepower	8.615 hp. per ton milled
Total connected kilowatts	6.42 kw. per ton milled

Current cost:

Total tons milled (one month) 22,016

Current consumption 124,320 kw.-hr.

$$\frac{124,320}{22,016} = 5.64 \text{ kw-hr. per ton milled}$$

Current cost was $0.0136 per kilowatt hour.

Power cost was $0.0136 × 5.64 = 7.68 cents per ton milled.

Crushing Cost.—The cost of crushing varies from about 9 to 12¢ per ton milled, depending primarily upon tonnage crushed. The figures in Table 25 represent about average when crushing 40 to 50 tons per hour.

Table 25.—*Crushing Costs*

	Cost per Ton	Per Cent of Total		Cost per Ton	Per Cent of Total
Hard iron	0.0429	44.5	Elevator cups and belt	0.0022	2.3
Power	0.0263	27.3	Screen jackets	0.0012	1.2
Maintenance and repairs	0.0100	10.4	Water supply	0.0009	0.9
Operating labor	0.0070	7.3	Lubrication	0.0001	0.1
Supervision	0.0033	3.4			
Repair labor	0.0025	2.6	Total	$0.0964	100.0

Table 26 gives costs for "hard iron," and, for comparison, steel consumptions when using manganese steel for crusher jaws and roll shells. The present price for "hard iron" is 2.75 cents per lb., which gives a total iron cost of 4.29 cents. This represents about as low a figure as can be anticipated.

Table 26.—*Costs for Hard Iron and Steel*

	Hard Iron		Steel	
	Lb. per Ton Crushed	Per Cent of Total	Lb. per Ton Crushed	Per Cent of Total
Primary crushing	0.25	16.1	0.0498	12.9
Secondary crushing	0.35	22.4	0.0988	25.7
Regrinding	0.96	61.5	0.2352	61.4
Total	1.56	100.0	0.3838	100.0

While there is a close agreement in the percentage distribution of the hard iron and the steel, the consumption of the former is 4.07 times that of steel. With hard iron at 2.75¢ this requires 11.2¢ steel to equalize costs, disregarding savings to be gained by increased running time and decreased labor for making replacements when using steel.

Concentrator Costs.—Analysis of the cost of concentrating in one plant disclosed the figures given in Table 27. These serve to give an idea of the caution with which the elimination of gravity concentration in favor of ball milling and flotation must be approached. An appreciable lowering of the jig or table tailing would be required to justify the increased operating cost and initial cost of plant necessary for such elimination; too much so to justify such a procedure from the standpoint of the economics involved with the spread that now exists, or should exist, between the gravity and flotation tailings.

TABLE 27.—*Concentrator Costs at One Plant*

	Cost per Ton Mill Feed	Cost per Ton Feed to Department
Jig mill department	$0.0654	0.0675
Table department	0.0411	0.1560
Ball-mill and flotation department	0.1124	0.5619
Tailing disposal	0.0151	0.0155
Total concentrating cost	$0.2340	

General Expense.—No attempt is made in our local accounting methods to proportion "overhead" or general expense against the mine and mill, this being carried in one general account.

Supplemental to the above data and subsequent to their compilation, one of the larger mining companies has been kind enough to submit its total operating costs for the year 1933 at one of its larger operations for publication in this paper, and it is herewith given in its entirety, showing both mining and milling as well as overhead (Table 28).

TAILINGS RE-TREATMENT

The district has seen a steady growth recently in milling operations on the tailings previously discarded from mills running mined ore. About 18 of these tailings mills are in operation at the present time, treating about 12,000 tons and producing about 175 tons of zinc concentrates every 24 hr. This represents about 20 per cent of the total production of the district. Only small amounts of lead concentrates are made. About one-third of the zinc concentrates are made by concentration on tables and the remainder by flotation.

Three principal reasons underlie the presence in the district of tailing piles rich enough to permit a profitable re-treatment operation: (1) inefficient mills used in the early stages of ore-dressing development for the district, many of which had no flotation plants, (2) overloaded mills, and (3) lack of proper mill control and supervision. The overloading of mills was caused mostly by the pressing of production during the heydey of the district, when concentrate prices were high. The mills were inadequate in capacity to withstand efficiently the demands for production.

TABLE 28.—*Typical Cost Sheet of Large-scale Operation*

FOR THE YEAR 1933

Tons ore hoisted (dirt)	169,395
Tons zinc concentrates produced	14,132.095
Coarse	10,521.82
Flotation	3,610.275
Tons lead concentrates produced	1,778.75
Recovery, per cent	9.39
Operating cost per ton ore	$1.091

Total Mining and Milling Cost	Per Ton Ore
Mining	$0.540
Milling	0.208
Shipping and hauling	0.001
Water, power and light[a]	0.158
Mine general expense	0.072
Prospecting	0.005
Administrative and general expense	0.107
Total operating cost	$1.091

Milling Operation	Cost per Ton Ore
Operating labor:	
Grizzly men	$0.003
Crusher feeder	0.007
Jig men and helpers	0.028
Table men and helpers	0.006
Flotation men and helpers	0.009
All other milling operating labor	0.007
Total operating labor	0.060
Flotation reagents	0.015
Extending tailings equipment	0.005
Belting, lubricants and supplies	0.015
Repairs to mill bldgs., bins, grizzly, etc	0.011
Repairs to machinery and equipment shop expense	0.102
Total milling operation except power	$0.208

[a] One-half of this item estimated chargeable to milling and one-half to mining.

Tailing-mill practice, with a few exceptions, follows closely in principle the scheme of milling mine ore, which has been discussed previously.

Mill feed is provided by means of a dragline, shovel, truck or conveyor or some combination thereof. Feed is stockpiled or stored in a hopper ahead of the mill. The flow sheet of a modern tailing mill proper parallels closely that of an up-to-date mill treating mine ore, except that the jaw crusher, primary rolls and cleaner jig are eliminated. The tailings treated do not contain mineral coarse enough for concentration on a cleaner jig.

Net recoverable values in the tailings treated currently vary between wide limits. Under the present marketing conditions the variation is approximately between 20¢ and 50¢ per ton. Out of this small sum must come all expenses of the operation, as well as amortization.

The narrow margin of values within which the tailing-mill operator must confine his operating expense makes necessary the very best of operating efficiency.

Costs.—A high amortization rate on plant investment is a major problem confronting the tailing-mill operator. Operation of a mill generally extends over a period of two to five years, necessitating amortizing charges of from 3 to 10¢ per ton of tailings treated. In order to minimize this charge, operators are inclined to group large tonnages of tailings (three to five million tons) for treatment over one mill of large capacity.

Operating costs of tailing mills, including cost of handling the mill feed, and excluding amortization, vary over the wide range of from 15¢ to 45¢ per ton of tailings treated.

As a rule the milling cost goes hand in hand with the recovery of mineral; i.e., the lower the recovery, the lower the cost, the lower recovery being due either to leaner tailings for mill feed, which can be run at a higher tonnage rate per concentrator unit, or to production of higher tailings. The daily cost of operating jigs, tables, elevators, etc., is much the same, irrespective of the tonnage handled; hence the greater the tonnage, the less the cost per ton treated. Low-cost and high-cost tailing mills also differ in their respective flow sheets. The low-cost mills have almost negligible amounts of grinding equipment that entail major costs for power and replacements.

TABLE 29.—*Cost of Operation at Two Tailings Mills*

Department	Mill A		Mill B	
	Cost per Ton, Cents	Remarks	Cost per Ton, Cents	Remarks
Loading	2.17[a]	By shovel	2.70[a]	By shovel
Hauling	2.95[a]	By truck	2.88[a]	By truck
Milling (labor and power)	12.98[b]	Minor grinding	19.29	Extensive grinding
General	2.87	Office expense, insurance, taxes, supervision	3.01	Office expense, insurance, taxes, supervision.
Total cost	20.97		27.88	

[a] Depreciation of equipment included.
[b] Before depreciation of mill equipment.

Table 29 is an abbreviated tabulation of milling costs of two tailing mills. Mill A treats 63.7 tons per hour and does moderate grinding in the circuit. Mill B treats 51.4 tons per hour and practices extensive grinding. Both mills are approximately the same size and entail nearly the same daily operating expense. Hence the difference in operating cost per ton lies principally in the milling of different tonnages with approximately the same work input.

Buying and Selling of Concentrates

Representatives of the various smelting companies using Tri-State concentrates maintain offices in the district. On Friday and Saturday of each week buyers confer with sellers by telephone and agree as to price, tonnage and date of delivery. In a few instances there are contracts calling for regular delivery of a certain tonnage, thus eliminating the weekly purchase. Otherwise no written contracts are used.

Both zinc and lead concentrate are now sold on a unit basis. Zinc concentrates are quoted on a basis of 60 per cent zinc content and lead concentrates on a basis of 80 per cent lead. However, zinc concentrates carry an arbitrary penalty of $1.00 per ton for flotation grades and an added penalty of $1.00 per unit for any grade below 56 per cent zinc content. Lead concentrates carry an arbitrary penalty, in addition to that computed on the unit price, of 50 cents per unit for grades below 80 per cent. The differential between gravity and flotation zinc concentrates, for a long time $3.00 per ton, was gradually reduced to nothing and only recently was restored to $1.00 per ton.

Allowable impurities in zinc concentrates are 3.0 per cent iron, 2.0 per cent lead and 1.5 per cent lime (CaO), and any excess beyond these limits is penalized at $1.00 per unit. Iron penalties are rare.

Differences between buyer's and seller's assays exceeding 0.6 per cent usually call for assay of the umpire sample, and the settlement is made on the middle assay. Otherwise the two assays are averaged as a basis for settlement. In some cases differences greater than 0.6 in the two assays may offset premiums against penalties in such a way as to avoid umpire. The umpire sample, assayed by a chemist agreeable to both parties, is paid for by the party whose assay is farther from the settlement assay.

On the day before delivery, box cars are ordered to be set on a railroad switch near the mine. These are loaded by the buyer's trucks, using hand labor for handling the concentrates from mill bin to truck and from truck to car. The trucks are weighed over the mining company's scales, and these weights generally are accepted by both buyer and seller, only rarely being altered by buyer's weights checked at the smelter. At the Eagle-Picher central mill, zinc concentrates are loaded into cars mechanically direct from a hopper.

When loading is completed, cars are sampled by a representative of the buyer in the presence of the seller's representative, using a "gun" sampler, which makes a number of vertical cuts about 2 in. in diameter throughout the car. The sample is hand-mixed on the spot and from it three small cans are filled, one for the buyer, one for the seller and one for umpire if needed. The latter sample remains in the custody of the buyer.

Trend of Practice

The general trend in milling practice in the Tri-State district is toward larger mills and machinery units. This practice has already reached considerable headway in mills treating mine ore and also in those treating tailings. Lower operating costs, longer life of operation and lower amortization rates are made possible thereby.

However, a wide divergence in the character of the ores to be milled, i.e., as to metal content, the ratio of the lead to the zinc and as to whether the ores are refractory to milling, such as the so-called "chatty" ore, or are "free-milling," may detract somewhat from the advantages of custom-milling, unless proper provision is made to mix ores of different physical characteristics and metal content in such a way as to provide a fairly uniform mill feed. Flexibility of flow sheet is necessary in order to make possible the most economical procedure with all classes of ores milled. Too rigid a flow sheet, especially when designed to handle the most refractory ores, does not permit of the most economical procedure when handling free-milling ores.

When the mill feed is uniform in metal content and physical characteristics, within reasonable limits, and the proper flow sheet is employed, metallurgical results are enhanced and economies of operation are effected in large plants, or at least should be. The cost of amortization of plant does not enter into its design to the same extent as in small plants, this permitting selection of the most efficient equipment for the work to be done.

Opportunities exist for improvement of the jig middling circuits following a truer appreciation of the physical characteristics of each middling product. Rather than combining two or more middling products of entirely different physical characteristics into one product for re-treatment and then subjecting this combined product to the procedure necessary for the recovery of the most refractory portion of such product, as much as 50 per cent of this total middling product, if further treated as originally segregated, might be made to yield equally good metallurgical results at an appreciable saving in operating costs. The solution of this problem does not lie in the adoption of what is the simplest procedure or the most expedient when designing a new plant, but in a consideration of all factors involved, such as cost of operation balanced against metallurgical results obtained and initial cost of plant. While simplicity of flow sheet is always desirable, this cannot be justified on the basis of increased operating costs, unless the added metal recovery offsets such increase, especially when such simplification does not decrease the initial cost of plant, as in adding grinding and flotation equipment in order to eliminate concentrating tables. Future practice in this connection is going to lie in utilizing the primary concentration of the refractory middlings grains, or chats, effected by the rougher jigs, and the isolation of this product for separate regrind to the point of unlocking its values before contaminating the other jig middling products, which are of free-milling character, and which can be made to yield very satisfactory results by secondary gravity concentration on tables. Unsatisfactory table tailings produced in the past are traceable to improper procedure in the preparation of the jig middling products for table feed, rather than to any inability of the tables to perform satisfactorily when given a properly prepared feed.

In connection with the primary concentration of the refractory middlings, or chats, more attention is being given to the advisability of jigging for such chats on the primary roughers in such a manner as will permit of the production of higher middlings and greater elimination of tailings by the primary roughers. A correlation between tailing mills and mills handling mine ores conclusively proves that when jigging for the light chats, the jig duty (i.e., the tons handled per jig) should be at least one-half that when jigging for mineral, in order to accomplish the most economical results. A recognition and adoption of such a circuit, combining the desirable features of both mine and tailing mills, points the way to the most economical procedure for mills handling mine run of ores; that is, a high jig duty when jigging for mineral and decreasing this duty to at least one-half when jigging for chats. Such a procedure will result in lower rougher tailings, greater tonnage eliminated by the rougher jigs and a rougher-jig chat or middling the grade of which is high enough so that very little, if any, justification will exist for secondary chat-roughing jigs.

Improvement in tabling practice will be made by using two table circuits, one for a relatively lean and free-milling sand and one for a chatty sand consisting of reground jig middlings.

Advances in flotation practice lie in using cleaner circuits to smooth out the finishing stage of the production of concentrates and eliminate much circulating mineral in the rougher cells.

More attention will be paid to the degree of grinding of various products in the mills. Middlings from jigs probably will receive finer grinding before subsequent treatment, and in some cases the jig feed will be reduced to a finer state before primary treatment.

There is a distinct trend to better equalized work for each machine in the mills. Jigs are not being burdened so heavily and produce better metallurgical results. Crushing and grinding machines, particularly ball mills, are being loaded more to capacity and are giving a better accounting of themselves. Flotation plants are being enlarged to a point where an economical balance is reached between milling cost and the minerals recovered. Better correlation will be made between milling costs and metallurgical results by means of technical analyses of milling circuits.

SUMMARY

An analysis of the foregoing discussion discloses that while the flow sheets are involved they are justified and essential on the basis of the attendant economics. The chert gangue is very abrasive and resistant to grinding, and such a large percentage of the lead and zinc values is freed by a comparatively coarse grind and recoverable by gravity methods of concentration that it is very doubtful whether much simplication of flow sheets can be justified economically. The field for improvement lies more in a truer appreciation of the physical characteristics of the different middling products resulting from gravity concentration and following the proper procedure in the re-treatment of such products, in order to obtain the maximum net production. Probably in no other mining district does ore dressing call for the careful balancing of metallurgy and operating costs that it does in this district; and such considerations become very much involved at times in pointing to the proper procedure to be followed.

Improvements in metallurgy made in this district since 1924 have accounted for an increase of from 1 to 3 per cent, by weight, increased concentrate production, locally termed "recovery." This represents from 20 per cent to as much as 30 per cent increased recovery of the mineral in the mill feed. Such improvement has been due in large measure to the installation of flotation departments and ball mills, together with improved metallurgy in the existing jig and table departments, all brought about by proper technical supervision, analysis and control of milling operations.

While the present status of milling does not offer the same opportunities for further improvement as existed in the past, the continuance of competent technical supervision is necessary to maintain maximum milling efficiencies, and to effect such further improvements in metallurgy and operating costs as are possible.

Also, while an analysis of the results does not point to any further material increase likely in the economic extraction of the mineral, it does clearly point the way to lower operating costs, some of which are already being realized in the district, as follows:

1. By 24-hr. operation, instead of single shift, resulting in a reduction in power costs of some 19 per cent, amounting to 1.5¢ to 2.5¢ per ton milled.
2. By increased tonnage milled, resulting in lower operating labor costs of from 4¢ to 15¢ per ton.

3. By substitution of steel for hard iron, resulting in lower operating costs due to decreased labor costs for making replacements, and to increased running time or tonnage milled when using steel.

4. By erecting more substantially built mills, permitting of lower maintenance costs of building and equipment.

5. Through increased tonnages discarded as gravity tailings, when possible to do so, even at a sacrifice of the grade of tailings produced, if the added recovery from subsequent re-treatment does not more than offset the increased cost due to such re-treatment.

6. By substitution of more efficient equipment for that being used, when its higher initial cost can be justified on the basis of the amortization costs involved.

When due regard is given to all the factors involved, total milling cost not exceeding 25¢ per ton should be obtainable in mills handling from 150 to 200 tons per hour.

Proper technical control and analysis of the metallurgical results obtained, correlated and balanced against the attendant economics, is the most vital and essential factor in obtaining the acme in metallurgical efficiency combined with the minimum operating costs. This factor, while it is the very essence of the problem, now frequently receives the least consideration, although the large tonnages milled more adequately justify it here than in most other mining districts.

Acknowledgments

Acknowledgment is made with appreciation to the companies that have contributed metallurgical data appearing in the paper. These include the St. Louis Smelting and Refining Co., with which Mr. Burris was associated for five years as the mill superintendent; the Evans-Wallower Lead Co., the Interstate Zinc and Lead Co., the Vinegar Hill Zinc Co. and the Century Zinc Co., the Tri-State Zinc, Inc. and the Missouri Mining Co., the Eagle-Picher Mining and Smelting Co., and the Commerce Mining and Royalty Co. for permission to publish the flow sheet of its Bird Dog mill. Also, appreciation is expressed to the Tri-State Zinc and Lead Ore Producers Association for the time and generous assistance of its office staff in the preparation of the manuscript for publication.

Nkana Mine Concentrator

By L. P. Durham*

During the early mine development period, 1929 and the first part of 1930, a 300-ton pilot-plant concentrator was built at Nkana mine of the Rhokana Corporation, Northern Rhodesia. This plant operated from May 26, 1930 until Oct. 26, 1931, treating 101,080 tons of Nkana ores and producing 4372 tons of concentrate.

The plant was built, primarily, as a test unit to determine the grinding characteristics, floatability, etc. of the Nkana ores and other ores of the Rhodesian copper belt and to supply sufficient data on which to base the design of a 5000-ton concentrating plant at Nkana mine. The test unit operated wholly on development ores and no attempt was made to grade or select the ores from any section of the mine. The average assays for the 17 months of operation are given in Table 1.

Table 1.—*Pilot-plant Operation for the Years* 1930 *and* 1931

	Total Cu	Oxide Cu	Fe	Co	S	Insol.
Mill feed	2.63	0.07	2.9	0.062	0.93	66.7
Concentrate	54.79	0.55	7.9	0.520	19.8	14.4
Tailings	0.25	0.04	2.4	0.029	0.10	69.6

The first ground was broken for the 5000-ton concentrator during June, 1930, and the construction of the plant was completed in November, 1931. The plant started on Dec. 10 and has operated continuously since that date.

The whole of the concentrating plant is compact and closely connected, yet with sufficient space for expansion of all sections up to 20,000 to 25,000 tons per day capacity.

Mining operations are carried on along a length of about four miles on the easterly limb of the Nkana syncline. This length includes the North orebody, from which all production has thus far originated, and the Mindola orebody, now being developed for production.

The ore-bearing measures in the North orebody are leached for some 75 ft. below the surface; the oxidized zone varies in vertical extent from 100 to 150 ft. Practically all the ore below the 200-ft. level is sufficiently

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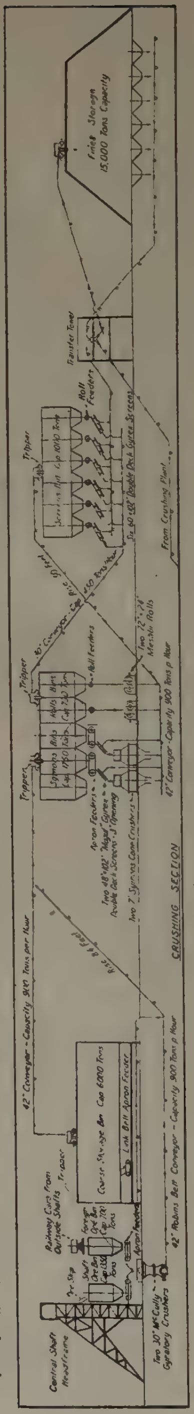
* Concentrator Superintendent, Rhokana Corporation, Limited, Nkana, Northern Rhodesia, Africa.

low in oxide copper to make possible treatment by normal sulfide flotation methods, and the oxide content decreases to negligible values below 350 ft. The ore consists of a gangue rock of more or less silicified shales, through which are disseminated bornite, chalcopyrite, chalcocite, carrollite and covellite. Typically, the secondary copper minerals decrease and the primary minerals increase with depth. All the ore treated to date has been extracted from above the 600-ft. level.

The ore is comparatively hard and abrasive, causing a moderate wear in both the crushing and grinding sections of the plant. The steel consumption is not excessive, however, and as the Nkana shops are equipped to make and finish both iron and steel castings, the cost per ton is low compared with usual copper-plant practice. The ore does not require exceedingly fine grinding: 62 to 65 per cent minus 200 mesh gives satisfactory metallurgical results; therefore the plant tonnage rate is considerably greater than the original estimated tonnage.

The flow sheet (Fig. 1) shows the flow through the entire plant. The Nkana concentrator follows closely generally accepted practice in copper flotation plants, so the following paragraphs will be largely confined to points of special interest.


Fines Storage 15,000 Tons Capacity
Transfer Tower
Roll Feeders
Tripper
Six 60"x108" Double Deck Gyrex Screens
From Crushing Plant
Rise 9 Feet
Conveyor
Two 72"x24" Mesabi Rolls
Roll Feeders
Tripper
Rolls Bins
Symons Bins
Tripper
Apron Feeders
Two 48"x102" "Niagara" Gyrex Double Deck Screens
Two 7' Symons Cone Crushers
42" Conveyor - Capacity 900 Tons p Hour
CRUSHING SECTION
42" Conveyor - Capacity 900 Tons per Hour
Rise 8 Feet
Coarse Storage Bin Cap 6000 Tons
Link Belt Apron Feeder
Tripper
Railway Cars from Outside Shafts
Foreign Ore Bin
Shaft Ore Bin
Ore Skip
Apron Feeders
42" Robins Belt Conveyor - Capacity 900 Tons p Hour
Central Shaft Headframe
Two 30" McCully Gyratory Crushers

Crushing Section

The crushing plant has three stages of crushing. Ore normally is received direct from the mine through the central shaft and shaft bin. Ore from other shafts on Nkana mine, or from other mining properties on the copper belt, is delivered to the primary crushing section through the standard-gage railway system and foreign-ore bin. Each crusher is fed from both bins through large apron feeders.

The Mindola mine, which is 4.34 miles from the Nkana concentrator, will have a similar shaft, shaft bin and coarse-crushing equipment. Following the one stage of crushing the ore will be delivered to the railway trucks through a conveying system and a 5000-ton loading bin. This ore, when delivered to the concentrator, will pass through a 5000-ton receiving bin. The ore will be fed through 18 flat sliding gates, a traveling apron feeder and one short 42-in. conveyor, on to a conveyor delivering to the secondary crushing plant.

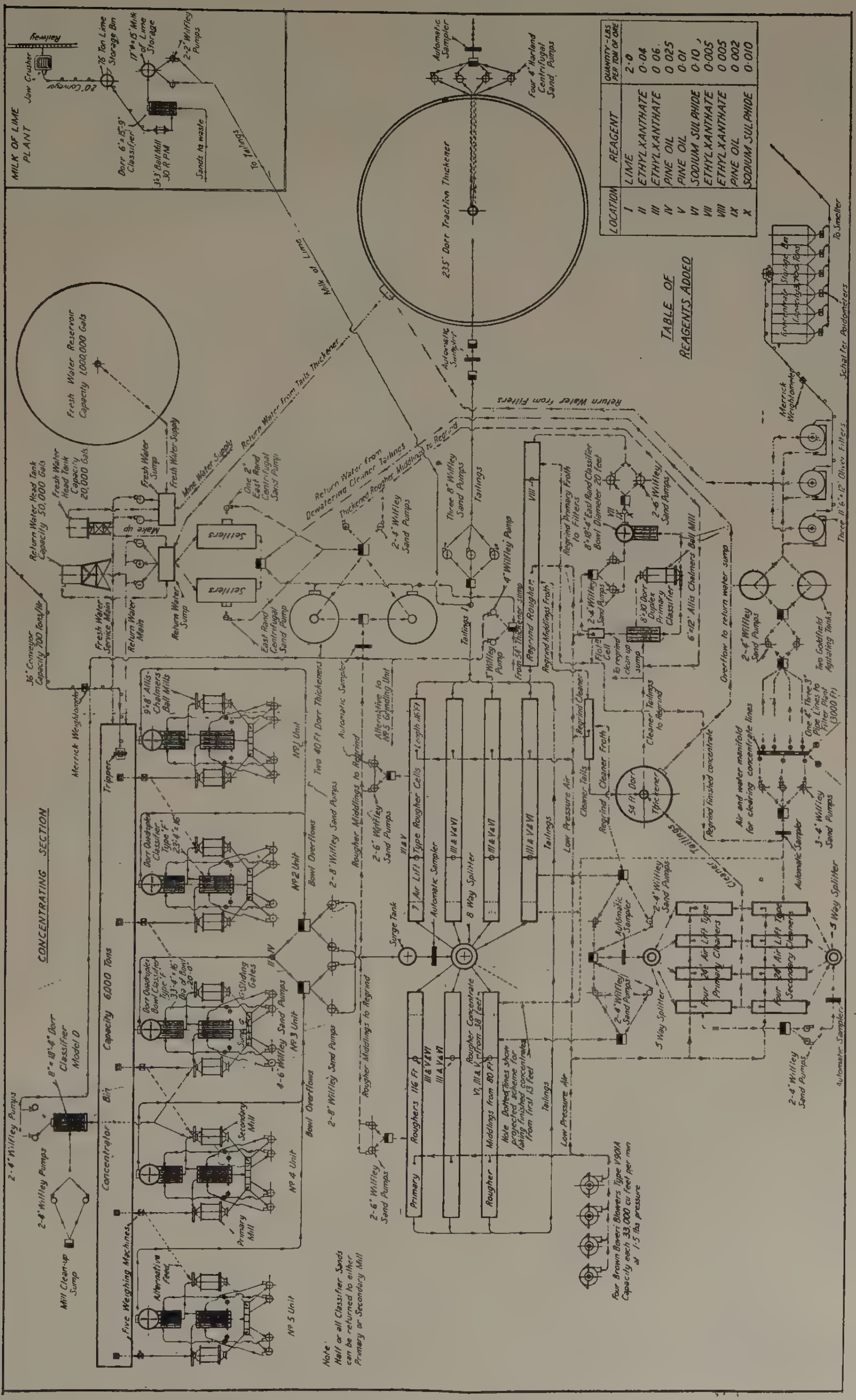

MILK OF LIME PLANT
Reamley
Jaw Crusher
20' Conveyor
75 Ton Lime Storage Bin
17'6"×15' Milk of Lime Storage
2-2" Wilfley Pumps
Dorr 6'×15'9" Classifier
5'×3' Ball Mill 30 R.P.M.
Sands to waste
To Tailings
Milk of Lime
Fresh Water Reservoir Capacity 1,000,000 Gals
Return Water from Tails Thickener
235' Dorr Traction Thickener
Automatic Sampler
Four 4" Harland Centrifugal Sand Pumps
TABLE OF REAGENTS ADDED
To Smelter
Schaffer Poidometers
Concentrate Storage Bin
Merrick Weightometer
Three 11'6"×12' Oliver Filters
Return Water from Filters
Return Water Head Tank Capacity 50,000 Gals
Fresh Water Head Tank Capacity 20,000 Gals
Fresh Water Sump
Fresh Water Supply
Mine Water Supply
Make up
Return Water Sump
Fresh Water Service Main
Return Water Main
36" Conveyor Capacity 700 Tons/Hr
Settlers
One 2" East Rand Centrifugal Sand Pump
East Rand Centrifugal Sand Pump
Return Water from Dewatering Cleaner Tailings
Thickened Rougher Middlings to Regrind
2-4" Wilfley Sand Pumps
Three 8" Wilfley Sand Pumps
Tailings
4" Wilfley Pump
3" Wilfley Pump
From 54 ft Thickener sump
Regrind Rougher
VIII
Regrind Middlings Froth
Regrind Primary Froth to Filters
8'×18'4" East Rand Classifier Bowl Diameter 20 Feet
VII
IX
2-6" Wilfley Sand Pumps
2-4" Wilfley Sand Pumps
8'×30' Dorr Duplex Primary Classifier
6'×12' Allis Chalmers Ball Mill
Flota Cell
To regrind clean up sump
Regrind Cleaner
Cleaner Tailings to Regrind
Overflow to return water sump
2-6" Wilfley Sand Pumps
Two Goldfield Agitating Tanks
One 4" Three 3" Pipe Lines to Filter Plant (3000 Ft)
Air and water manifold for clearing concentrate lines
Regrind finished concentrate
3-4" Wilfley Sand Pumps
Automatic Sampler
5 Way Splitter
Four 24' Air Lift Type Secondary Cleaners
Four 24' Air Lift Type Primary Cleaners
2-4" Wilfley Sand Pumps
Automatic Sampler
5 Way Splitter
Tailings
Cleaner Tails
Cleaner Froth
Regrind
54 Ft Dorr Thickener
Low Pressure Air
Tailings
CONCENTRATING SECTION
Merrick Weightometer
Tripper
9'×8' Allis-Chalmers Ball Mills
Dorr Quadruplex Classifier Type "F" 23'4"×16'
Dorr Quadruplex Bowl Classifier Type "F" 33'4"×16' Dia of Bowl 20'0"
Sliding Gates
Sump
Secondary Mill
Primary Mill
Alternative Feed
Nº 1 Unit
Nº 2 Unit
Nº 3 Unit
Nº 4 Unit
Nº 5 Unit
Two 40 Ft Dorr Thickeners
Automatic Sampler
Alternative to Nº 5 Grinding Unit
Bowl Overflows
2-8" Wilfley Sand Pumps
4-6" Wilfley Sand Pumps
Rougher Middlings to Regrind
2-6" Wilfley Sand Pumps
Air Lift Type Rougher Cells (Length 36 Ft)
VII & V
III & V & VI
8 Way Splitter
Surge Tank
Automatic Sampler
Primary
Roughers 116 Ft
Rougher
Middlings from 80 Ft
Rougher Concentrate VI, III & V from 36 Feet
Note: Dotted lines show projected scheme for taking finished concentrates from first 13 Feet
Capacity 6000 Tons
Concentrator Bin
8"×18'4" Dorr Classifier Model D
2-4" Wilfley Pumps
2-4" Wilfley Pumps
Mill Clean-up Sump
Five Weighing Machines
Note: Half or all Classifier Sands can be returned to either Primary or Secondary Mill
Four Brown Boveri Blowers Type V901A Capacity each 33,000 cu feet per min at 1.5 lbs pressure

TABLE OF REAGENTS ADDED

LOCATION	REAGENT	QUANTITY—LBS PER TON OF ORE
I	LIME	2·0
II	ETHYL XANTHATE	0·04
III	ETHYL XANTHATE	0 06
IV	PINE OIL	0 025
V	PINE OIL	0·01
VI	SODIUM SULPHIDE	0·10
VII	ETHYL XANTHATE	0·005
VIII	ETHYL XANTHATE	0 005
IX	PINE OIL	0 002
X	SODIUM SULPHIDE	0·010

FIG. 1.—FLOW SHEET OF NKANA CONCENTRATOR.
(Capacity 9000 tons per 24 hours, December, 1934)

The secondary crushing plant consists of two 7-ft. Symons cone crushers and two 72 by 24-in. Mesabi-type roll crushers, all set in line under one crane way. All four crushers discharge on to a single 42-in. conveyor, which delivers the crushed product to the screening plant.

Normally the McCully crushers are set to 6 in., the Symons crushers to ½ in. and the rolls to ¼ in. The double-deck Gyrex screens have 1¼-in. square-opening screen cloths on the upper deck and ⅜ by ¾-in. opening screen cloths on the lower deck. At present the top deck is used only to break up lumps of fines and to scalp off the coarser rock. Later it is proposed to return the top-deck oversize to the Symons crushers.

The crushing plant will deliver 500 to 600 tons per hour to the ball-mill bins, making a product as shown in Table 2.

TABLE 2.—*Screen Sizing, Ball-mill Feed*

MOISTURE IN FEED, 4.5 PER CENT

MESH	PER CENT WEIGHT	MESH	PER CENT WEIGHT
3	34.5	100	14.5
4	13.0	200	4.0
14	27.0	−200	7.0

Provision has been made in the secondary plant for an additional 7-ft. Symons crusher. With this machine installed the plant capacity will be 800 to 900 tons per hour.

GRINDING SECTION

Practically any desired flow can be obtained through the grinding section by opening or closing a few valves and sliding gates. (See Figs. 1 and 2.) Either or both mills to a section can be fed from the ore bins, and either or both mills can be operated with either or both primary and secondary classifiers. The sands from each classifier can be divided between the two mills at the will of the operator. Each unit is equipped

TABLE 3.—*Screen Sizing, Flotation Feed*

SOLIDS IN FEED 28 PER CENT BY WEIGHT

MESH	PER CENT WEIGHT	MESH	PER CENT WEIGHT
48	2.0	150	8.5
65	4.5	200	12.5
100	8.5	−200	64.0

TABLE 4.—*Steel Consumption*

	LB. PER TON
McCully crusher, concaves and mantles	0.0043
Symons cone crushers, mantles and bowls	0.0165
Roll shells and cheek plates	0.07
Ball-mill liners	0.155
Ball-mill steel balls	1.414

with a weighing machine, which indicates, integrates and records the tonnage rate.

The tonnage rate per unit is 1800 to 1850 dry tons per 24 hr. when making a finished product as shown on Table 3. Recent experiments on ball-mill speeds indicate that each unit tonnage rate can be increased considerably. Steel consumption is shown in Table 4.

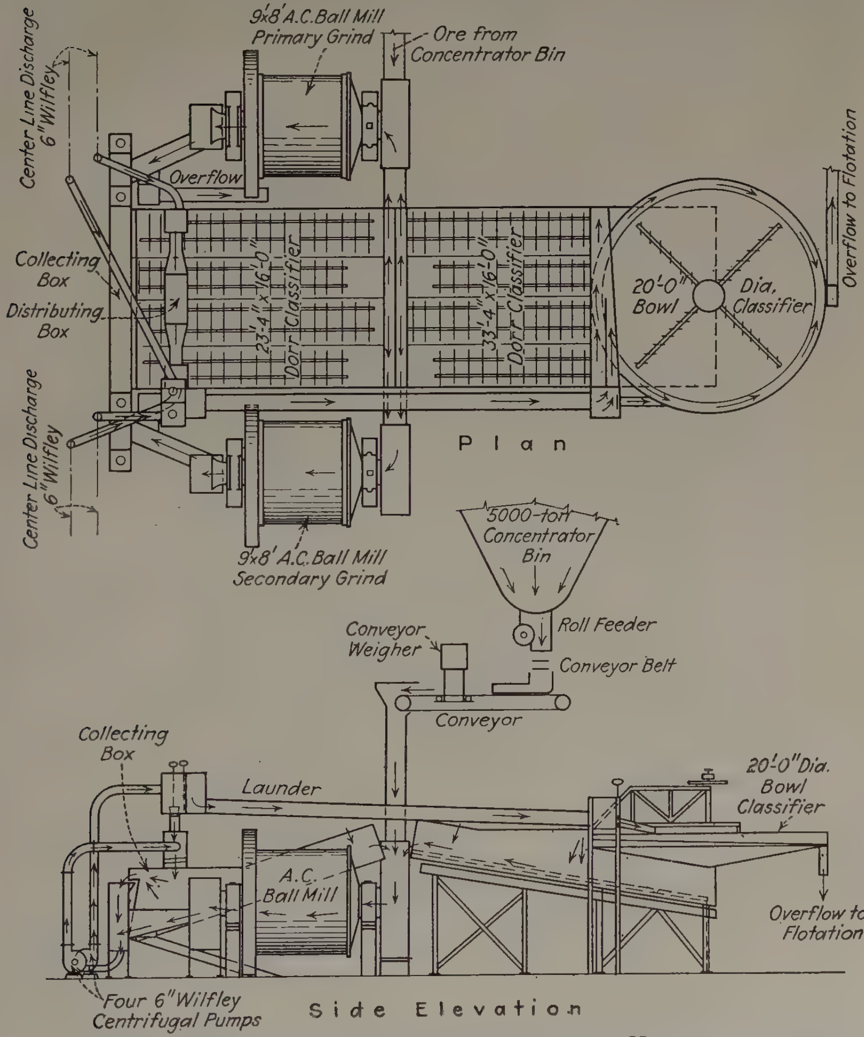


FIG. 2.—LAYOUT OF BALL MILLS AND CLASSIFIERS, NKANA MINE.

FLOTATION SECTION

The flotation section contains seven air-lift roughers (Fig. 3), 116 ft. long, each treating around 1500 tons of initial feed. The cleaner and recleaner cells are identical in construction to the rougher cells except for the width of the frothing chamber, which is 22½-in. wide instead of 15½ in., as shown.

The total air input for the whole of the flotation section, which includes 1126 ft. of air-lift cell, is around 100,000 cu. ft. per minute. The air is

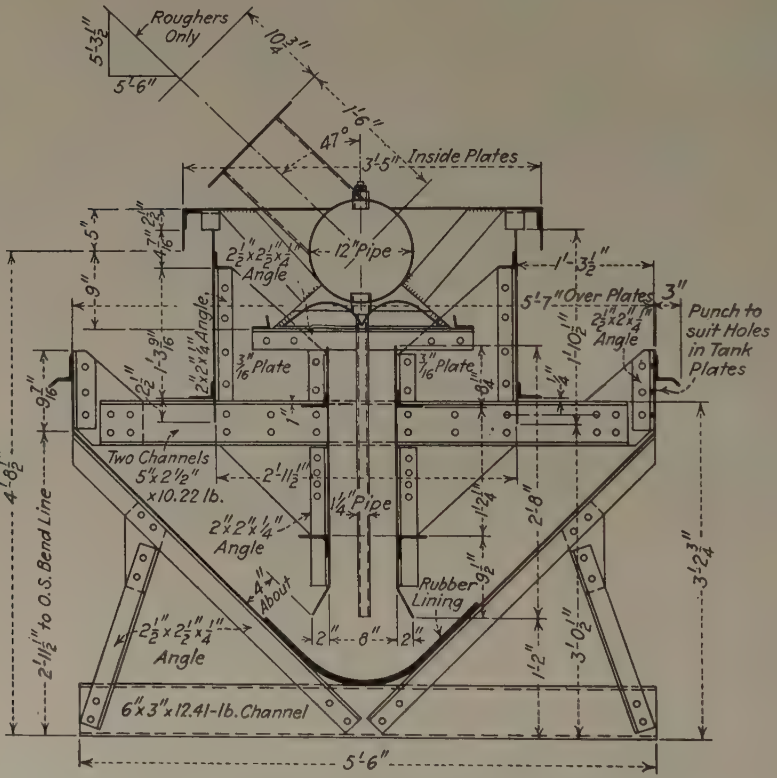


FIG. 3.—SECTION THROUGH ROUGHER MACHINE.

TABLE 5.—*Assay Values, Flotation Section*

	Percentages					
	Cu	CuO	Fe	S	Co	SiO₂
Mill feed	4.34	0.26	2.78	1.79	0.22	44.88
Concentrate	53.94	0.81	9.58	23.07	2.13	5.86
Tailings	0.56	0.22	2.15	0.16	0.072	47.86

	PER CENT
Total copper recovery	88.0
Sulfide copper recovery	91.8
Cobalt recovery	69.6

Ratio of concentration, 14.1 to 1

supplied by four blowers of the centrifugal turbo type, each delivering about 33,000 cu. ft. per minute at 1.50 lb. air pressure.

The total amount of ore treated to Aug. 1, 1934, was 4,176,000 short dry tons, of which the assay values and recovery are given in Table 5. Recent laboratory tests on Mindola ores indicate a higher recovery, 94 to 95 per cent total copper, and the most desirable concentrate for smelting. The ore grinds more readily than Nkana ores and fine grinding is not required because the mineral is not so intimately associated with the gangue.

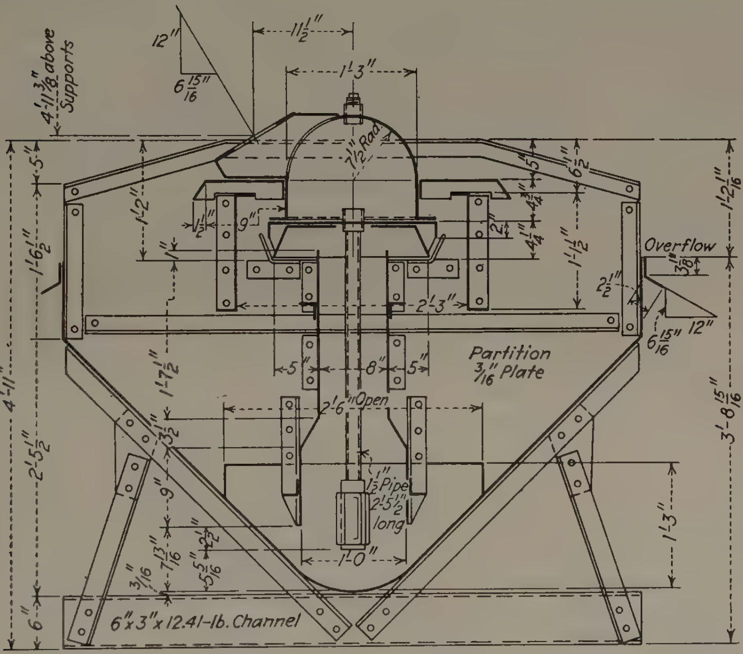


FIG. 4.—GENERAL SECTION, FLOTATION MACHINE.

Assays of concentrates produced from Mindola ores are as follows: Cu, 60.0 per cent; CuO, 0.17; Fe, 9.5; S, 22.7; Co, 0.10; SiO_2, 3.4; Al_2O_3, 1.10. Total copper recovery, 95.3 per cent.

REGRIND SECTION

During the early tests in the pilot plant it was found that a portion of the mineral in Nkana ores is finely disseminated chalcocite intimately associated with gangue. The bulk of the minerals, particularly from the central and south sections of the mine, is coarsely disseminated bornite with a smaller amount of chalcopyrite. This portion will readily float at a comparatively coarse grind, producing a satisfactory grade of concentrate and low tailings losses. The chalcocite ores, from laboratory tests and microscopic examination, has gangue-attached mineral when ground through 500 mesh or smaller. A comparatively low tailing can be obtained, however, from the rougher cells, providing all middling products are treated separately and not returned to the roughing circuit.

The present flow sheet at the concentrator is based largely on the early tests at the pilot plant, and tests carried out at the concentrator during 1932 and the first half of 1933. All middling products, including cleaner tails, recleaner tails and rougher middlings, are dewatered and reground in a grinding unit specially arranged for fine grinding. The product from the regrind section is floated separately and no middlings are returned to the rougher cells. All fresh water required for mill make-up water is added to the cleaner and recleaner cells for reagent dilution. This arrangement has reduced the tailings losses around 0.10 per cent copper and produces a concentrate more suitable for smelting.

TABLE 6.—*Assays for Period from Jan.* 1, 1934 *to July* 31, 1934
REGRIND UNIT AND NEW TYPE CELLS IN OPERATION

	Percentages					
	Cu	CuO	Fe	S	Co	SiO_2
Heads	3.73	0.18	2.72	1.57	0.20	43.38
Concentrate	52.15	0.70	10.92	24.15	2.07	5.44
Tails	0.40	0.12	2.22	0.12	0.06	45.77

	PER CENT
Total copper recovery	90.0
Sulfide copper recovery	92.6
Cobalt recovery	72.1

Ratio of concentration, 15.5 to 1

REAGENTS

All reagents, except lime, are fed by the disk and bucket type of feeder. Many reagent combinations have been tried both at the pilot plant and concentrator. The most satisfactory combination from the point of view of recovery, concentrate grade and economy is shown on Table 7. The presence of sodium sulfide in the roughing cells has reduced the tailings losses around 0.02 to 0.03 per cent Cu and lowered the xanthate consumption about 15 per cent.

The lime is fed to the tailings thickener as milk of lime; 2 lb. per ton fed to the mill tailings gives the desired results. Several tests have been made on the quantity required, and the points of feeding into the circuit. A low-grade lime (about 50 per cent available CaO) from a local lime quarry is delivered to the lime plant. The lime is crushed and ground by the equipment as indicated in Fig. 1, and the milk of lime is delivered to various points in the mill through 2-in. Wilfley pumps and 2½-in. pipe lines. The reject from the classifier is either run to waste or joins the pulp flow to the tailings thickener.

TABLE 7.—*Reagent Consumption*

FLOTATION SECTION

	LB. PER TON
Lime (50 per cent available CaO)	2.0
Ethyl xanthate to head of rougher cells	0.04
Ethyl xanthate to middling section of roughers	0.06
Pine oil to head of rougher cells	0.025
Pine oil to middlings section of roughers	0.01
Sodium sulfide to middlings section of roughers	0.10

REGRIND SECTION

Ethyl xanthate to head of 116-ft. rougher cell	0.005
Ethyl xanthate to middlings section of rougher	0.005
Pine oil to head of rougher cell	0.002
Sodium sulfide to head of rougher cell	0.01

FILTERING SECTION

Concentrates are delivered direct to the filtering plant from the recleaner cells at about 50 per cent solids without dewatering. Practically no trouble is experienced in pumping the concentrate through the 3000-ft. 3 and 4-in. pipe lines. Normally one 4-in. Wilfley pump and one 3 or 4-in. line is used. Two of the Oliver filters will readily handle

TABLE 8.—*Power Consumption*
JUNE, 1934

	Kilowatt-hour per Ton Ore		Kilowatt-hour per Ton Ore
Primary crushers	0.349	*Regrind Unit*	
Symons crushers	0.402	Ball mill	0.262
Rolls	0.541	Pumps	0.122
Screens	0.262	Low-pressure air supply	0.349
Conveying	0.279		
		Total	0.733
Total	1.833		
		Tailings disposal	0.496
Ball mills	7.507	Concentrate handling	0.141
Ball-mill auxiliaries	1.432	Concentrate filtering	0.325
Low-pressure air supply	2.243	Miscellaneous	0.347
Flotation pumps	0.925		
Water supply	0.576	Total	1.309
Total	12.683	Grand total concentrating	14.725
		Grand total crushing and concentrating	16.558

the concentrates from the whole of the plant, delivering the concentrates directly to the smelter conveying system at 7 to 8 per cent moisture.

Tailings Disposal

The tailings, after passing through a 235-ft. thickener, flow through a 3100-ft. flume to the tailings pond. (See Fig. 3.) An earth dam across one of Rhodesia's dambos, or swamps, provides storage space for about 20,000,000 tons of tailings. Approximately one ton of water is lost for each ton of tailings.

Concentrating Operations at the Roan Antelope Copper Mines Limited

By J. W. Littleford*

The Roan Antelope Copper Mines Ltd. are situated in the north central part of Northern Rhodesia, at an altitude of 4000 ft. and 13° south of the Equator. The town site (Luanshya) is well situated and enjoys among other things a really temperate climate for 10 months of the year. The two spring and summer months, October and November, while hot and humid, do not bring any great discomforts as compared with many other mining camps. Strictly modern brick homes, with electric lights, purified water, telephone and water-borne sewage, add to the comfort of the employees.

A rainfall of approximately 60 in. per year furnishes ample water for both industrial and domestic purposes. The rainy season generally begins in October and lasts through until March. As much as 3 to 4 in. is recorded in 24 hr., but this offers no difficulties because the runoff is very rapid.

The property is connected by a 24-mile branch line from Ndola with the main line of the Beira-Mashonaland and Rhodesia Railways Ltd. Practically all supplies from overseas are transported over this line from Beira, 1743 miles distant on the east coast in Portuguese East Africa. The mine output also leaves Africa from this port. Passenger service is principally via Cape Town or Durban, a distance of about 2100 miles to either port. The Imperial Airways maintain a service from Cape Town to Croydon, taking 9 days for the trip. Connections can be made with this service at Broken Hill, 150 miles south of Luanshya, by either rail or automobile. Thus, after several days of travel through virgin bush, a visitor arrives at a typically modern town site in the heart of Africa. Every development in mining, milling and smelting practice is used to advantage.

Plan of Mill

The practice in the three principal phases of the production of copper from the ores of this company have been published from time to time. Milling practice, however, is continually changing and what may be

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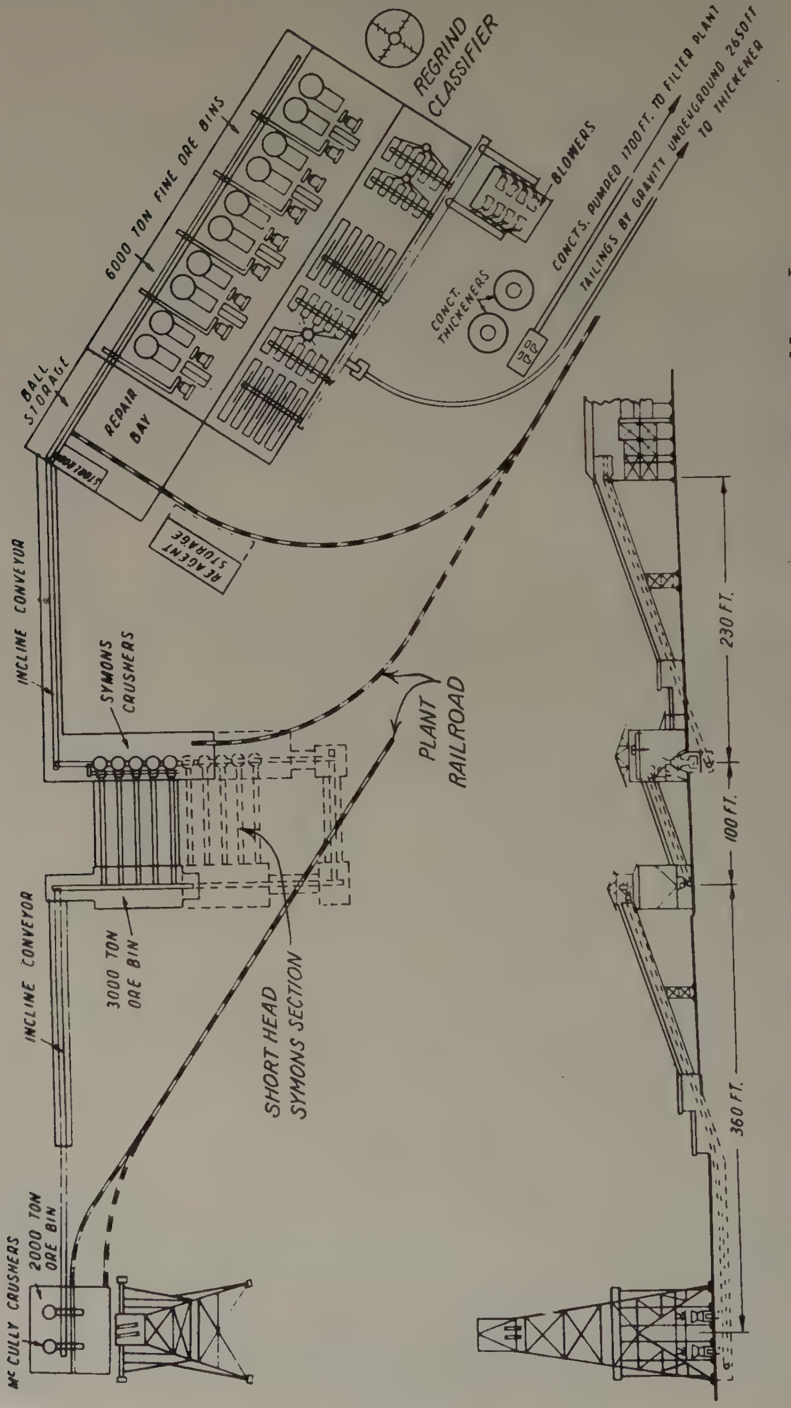

FIG. 1.—GROUND PLAN OF CONCENTRATION PLANT, ROAN ANTELOPE COPPER MINES, LTD.

described today is obsolete tomorrow. Therefore it is the purpose of this paper to give a general outline of present practice and probable developments.

The flow sheet of the Roan Antelope concentrator has undergone several changes during its three years of operation, and if present indications prove up will pass through several more in the near future. The ground plan of the concentrator (Fig. 1) indicates the location of the several milling units in relation to the Beatty shaft, which is the main hoisting shaft at present. The coarse-crushing and fine-crushing departments are closely connected to the mill proper, making for very close cooperation between these units. The skips from the Beatty shaft discharge directly into the coarse-ore bins at the primary crushing plant. The concentrate-filtering plant, tailings-thickening plant and tailings dam are 1700 ft., 2500 ft. and 3500 ft. respectively in a southeasterly direction from the mill.

In order to take advantage of cheap transportation of concentrates by pumping, the filter plant is located close to the smelter storage bins for flux, charge and reverts. The tailings thickener was so placed as to obtain a gravity flow from the mill and still be adjacent to the dam, thereby making use of only one pumping operation to complete the disposal of tailings.

Ore Treated

The ore-bearing series of the Roan Antelope consists of dark gray-green, gray and sometimes brownish metamorphosed argillaceous shales and sandstones. Sandy partings, stringers of quartz, orthoclase, pegmatite and seams of calcite are often present. Shale is the principal mineral-bearing rock, while chalcocite, bornite and chalcopyrite are the chief copper minerals present. The distribution of these is variable. In the eastern zone (that which is now being mined) chalcocite is dominant, whereas in the western area chalcopyrite and bornite predominate. The chalcocite is finely disseminated throughout the rock mass, and is rarely discernable with the naked eye.

Outcrop oxidation continues down to the water table (100 to 200 ft.). Below this level it still persists, but in diminishing quantities. The oxides usually are chrysocolla, malachite, cuprite and melaconite, while native copper and sooty-black manganese oxides (named in order of importance) are also present.

Typical mill feed assays show: total Cu, 3.24 per cent; oxide Cu, 0.29; SiO_2, 54.79; Al_2O_3, 17.97; Fe, 3.22; S, 0.95; CaO, 1.30; MgO, 4.78; Residue, 13.79. The oxide content of the mill feed was much higher at first, but is gradually diminishing, as a result of the mining program, which calls for stoping the upper (and hence more highly oxidized) levels first.

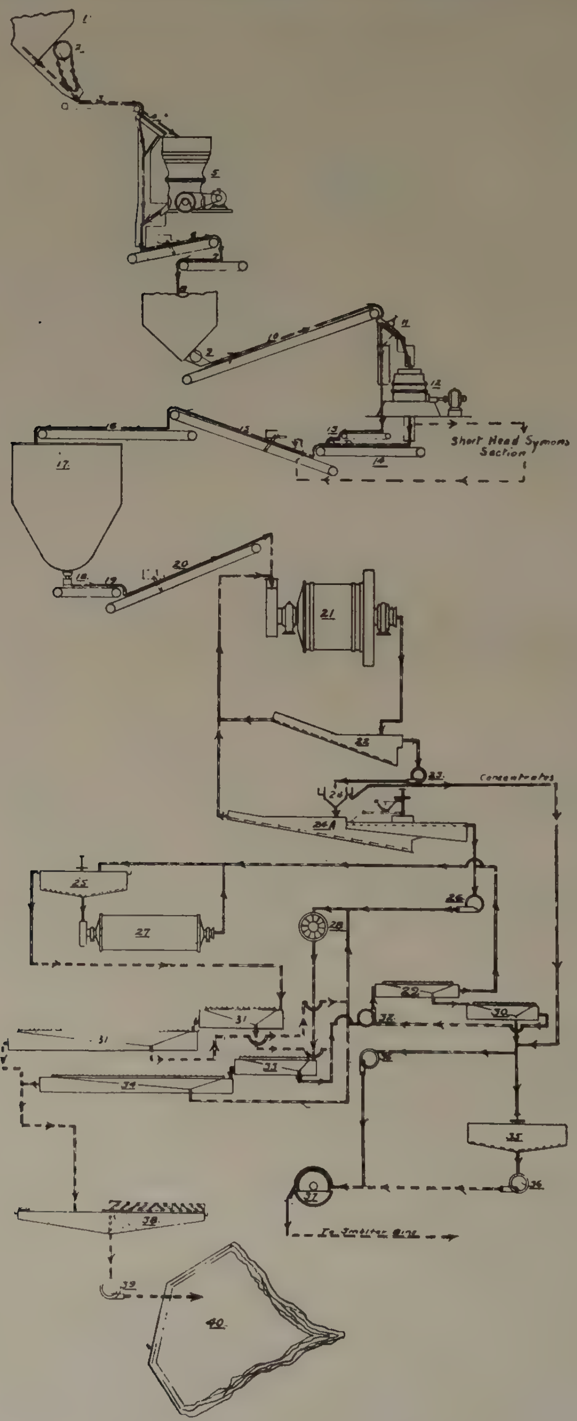

Fig. 2.—General flow sheet of concentration plant. For key numbers see opposite page.

Crushing and Grinding

From the standpoint of crushing and grinding the ore presents few problems. During the first two years of operation it could be classed as medium soft: it was easily crushed and caused very little wear on the crusher parts, owing to the preponderance of shale, which is not abrasive. As the mining has proceeded westwards, the ore has gradually become somewhat harder, with a corresponding slight increase in the wear on the crusher parts. Classification is also simple with present practice, and no difficulty is experienced in obtaining 90 per cent minus 200-mesh product with 30 to 32 per cent solids. A complete general flow sheet, giving all pertinent data, is shown in Fig. 2. While following generally accepted practice, there are a number of noteworthy features.

Coarse Crushing

Starting with the coarse-crushing plant, the ore as received from the mine has all passed 18-in. grizzlies, but, owing to the slabby nature of the rock, individual pieces at times will weigh 500 to 600 lb. The two six-chain Ross feeders furnish a very uniform feed, and it is seldom necessary to blast to loosen the rock that has arched over in the bins. Very fine ore, high in moisture, causes some trouble, but is easily loosened with a very small stream of high-pressure water. These feeders are followed by two belt conveyors, which discharge to two grizzlies. The

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Key Numbers

1. Beatty headframe bins, 2000 tons capacity.
2. Two 6-chain Ross feeders, 4¼ r.p.m., 10-hp. motors through Texrope and reduction gear.
3. Two 60 in. by 16-ft. conveyors, 37.5 ft. per minute, 5-hp. motors.
4. Two 6 by 10-ft. grizzlies, 4½-in. openings, hinged bottom end, coil springs top end.
5 Two 30-in. superior McCully crushers, 180 hp. motor drive through fourteen 1½ by 1-in. Texropes.
6 One 42-in. belt conveyor (No. 2) 380 ft. long with incline lift of 72-ft., 125-hp. motor.
7 One 42-in. belt conveyor (No. 3) 109 ft. long with automatic traveling tripper.
8. Symons storage bins, 3000 tons capacity.
9. Five 48-in. drum feeders.
10. Five 24-in. belt conveyors (Nos. 4A–E) incline lift of 27 ft., magnetic head pulleys.
11. Five 4 by 6-ft. type 60 Hum-mer screens, V64 vibrator, ½ by 1¼-in. screen openings at 37°.
12. Five 5½-in. Symons cone crushers direct connected to 150-hp. motors.
13. One 30-in. collecting conveyors and 30-in. cross conveyor for Hum-mer screen undersize.
14. One 42-in. belt conveyor (No. 5) 85 ft. long.
15. One 42-in. belt conveyor (No. 6) 273 ft. long—incline lift of 75 ft.
16. One 42-in. belt conveyor (No. 7) 375 ft. long with automatic traveling tripper.
17. Fine ore bins—capacity 6000 tons—1200 tons per unit.
18. Thirty 18-in. roll feeders—6 feeders per unit.
19. Five 20-in. belt conveyors (No. 8) 46 ft. long—collecting from roll feeders.
20. Five 20-in. belt conveyors (No. 9) 60 ft. long with incline lift of 17 ft.—weightometer on each.
21. Ten No. 98 Marcy ball mills direct connected to 400-hp. syn. motors.
22. Ten 6 by 20-ft. Dorr circulating classifiers—altered from five 12 by 20-ft. classifiers.
23. Fifteen 6-in. Wilfley sand pumps. Two operating—one spare, for each two mills.
24. Ten 12-ft. cascade flotation machines.
24A. Ten Dorr quadruplex bowl classifiers 12 ft. by 32 in. with 18-ft. bowls—10 and 5-hp. motors.
25. One 30-ft. Dorr thickener altered to 6 ft. in height—to classify regrind products.
26. Three 8-in. Wilfley sand pumps. Handlin bowl classifier overflow to flotation.
27. One 4 by 12-ft. Fraser and Chalmers ball mill for regrinding.
28. One weir type flotation feed distributor.
29 Five 22-ft. matless flotation machines—primary cleaners.
30. Five 16-ft. matless flotation machines—secondary cleaners.
31 One 22-ft. and one 60-ft. matless flotation machine for reground products.
32. Three 4-in. Wilfley sand pumps.
33. Ten 22-ft. matless flotation machines—primary roughers.
34. Ten 60-ft. matless flotation machines—secondary roughers.
} one set used as in 31
35. Two 35 by 10-ft. Dorr concentrate thickeners Provision made for by-passing.
36. Two 3-in. type "CA" Wilfley pumps—40-hp. motors—1700 ft. to filter plant.
37. Three 8 by 8-ft. Oliver filters.
38. One 250-ft. Dorr traction thickener.
39. Four 6-in. Wilfley sand pumps—125-hp motors.
40 Tailings dam.

load here reaches a peak of 900 to 1000 tons per hour per belt, depending on the size of the feed. All rock that has passed the underground grizzlies is handled without further reduction. The life of these belts is short, owing to large pieces of rock falling directly on to the belt rather than to ordinary wear. In spite of this, however, belts of good quality carry over 900,000 tons each. The cost of maintenance, which includes the cost of new belts, is less than 0.002 pence per ton.

The original grizzlies have been altered to take care of wet ore, which is encountered periodically and formerly caused considerable delay. The changes included spreading the bars at the lower end to leave a tapered opening between bars toward this end and mounting the upper end of the grizzly frame on heavy coil springs. The lower end of the frame is hinged. The impact of heavy pieces of rock falling directly over these springs sets up sufficient vibration to clear the grizzly, in addition to reducing the terrific shock on the grizzly bars. It is seldom necessary to shut off the feed to clean the grizzlies, whereas with the rigid type with parallel bars this was done several times per hour.

The two McCully gyratories, set to crush to 4½ in., have an hourly capacity of 500 to 750 net tons each with an initial feed of 750 to 1000 tons per hour, depending, of course, on the size and character of the ore. Only one crusher is in operation at a time, the other being held in reserve. Large thin slabs of rock often escape the action of the crushers and are passed on to the Symons plant. A 42-in. inclined conveyor, 380-ft. centers, takes the combined crusher product and grizzly undersize to the distributing conveyor over the Symons bins. The latter conveyor is equipped with a semiautomatic tripper for the distribution of ore to the different sections of the bin.

The plant as a whole operates very efficiently. It handles from 8000 to 10,000 tons, working on a basis of 1½ shifts per 24 hr. This includes ample time for cleaning up, oiling, etc. On one shift two men are needed for coarse crushing and conveying to the Symons bins. On the second shift the two men alternate between the coarse-crushing and fine-crushing plants. Cost per ton coarse crushed averages 0.03 shilling, distributed as follows: labor, 0.01s.; power 0.01s.; stores, etc., 0.01s.

Fine Crushing

The flow sheet of the fine-crushing plant is very simple. Ore is delivered from the coarse-ore bins to the five type 60 Hum-mer screens and five 5½-ft. Symons crushers by roll feeders and 24-in. conveyors, equipped with magnetic head pulleys. The rate of feed is regulated from the Symons operating floor by remote control to the roll feeders. The Hum-mer screens are set at an angle of 37° and are equipped with wire cloth of ½ by 1¼-in. mesh, which removes approximately 15 per cent of the original feed as undersize. Screens and crushers are in open circuit.

The Hum-mer screen undersize is of a very clayey nature and carries a large portion of the moisture in the ore. It was necessary to install an auxiliary conveyor to transfer this material to the cross conveyor under the Symons crushers, as it would not run in a chute placed at an angle. The moisture contained in the undersize is largely absorbed by the Symons product, making it easy to handle and yet practically free of dust.

The dust created in fine crushing is well handled by the dust-control system designed by George E. Lynch.[1] No difficulties have developed in the system.

As in the coarse-crushing plant, the wear on the crusher wearing parts has been light. Cone and bowl liners are good for over 1,000,000 tons of initial feed to each of the Hum-mer screens, or 850,000 tons of oversize product to each Symons crusher.

All cast-iron chute liners have been replaced with locally made grid-type liners in which the ore forms its own wearing surface by filling in the spaces between the grid bars.

The following is a typical screen analysis of mill feed: on 1.5 in., 1.8 per cent weight accumulative; on 0.371 in., 57.9; on 10 mesh, 84.0; on 48 mesh, 89.1; on 100 mesh, 90.2; on 200 mesh, 91.1.

Sizing tests on this ore are liable to be misleading in that the amount of oversize, while appearing excessive, is due to the fact that a great portion is in the shape of thin slabs.

The same number of Europeans are employed for fine crushing as for coarse crushing. A crusher foreman, one repairman and six operators comprise the entire coarse-crushing and fine-crushing European operating force; 30 natives are employed.

Cost distribution of coarse and fine crushing, and conveying up to the mill bins, follows: labor, 0.03s.; power, 0.03s.; maintenance, supplies, etc., 0.04s.; total, 0.10 shilling.

The installation of five 5½-ft. Symons short-head crushers to operate in closed circuit with screens is now under way and it is estimated that an increase of 200 tons per ball-mill day will be realized. This increased capacity will follow from the considerable reduction in the average ore size of the feed to the ball mills. The cost of reduction in the Symons will be cheaper also than with the less positive action in the ball mill.

Fig. 3 shows the flow sheet of the complete fine-crushing department.

The mill bin is of the catenary type with capacity of 6000 tons, which at present is sufficient for 16-hr. run with two mills per section in operation. Roll feeders driven through worm-gear reducers, and adjustable as to speed and chute opening, deliver mill feed to a cross conveyor which discharges on to an inclined conveyor delivering into the ball-mill scoop box. The inclined conveyors are equipped with Merrick weightometers. The feed can be held to very uniform rates and variations are seldom over 1 per cent.

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[1] G. E. Lynch: Dust Control an Economy Measure. *Eng. & Min. Jnl.* (1932) **133**, 606.

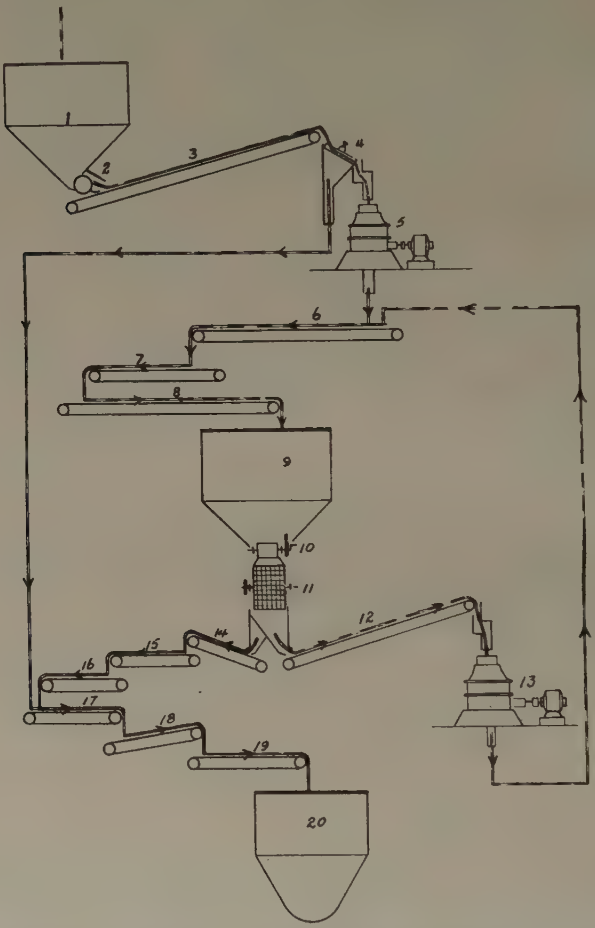


Fig. 3.—General flow sheet of Symons plant arranged for closed-circuit crushing.

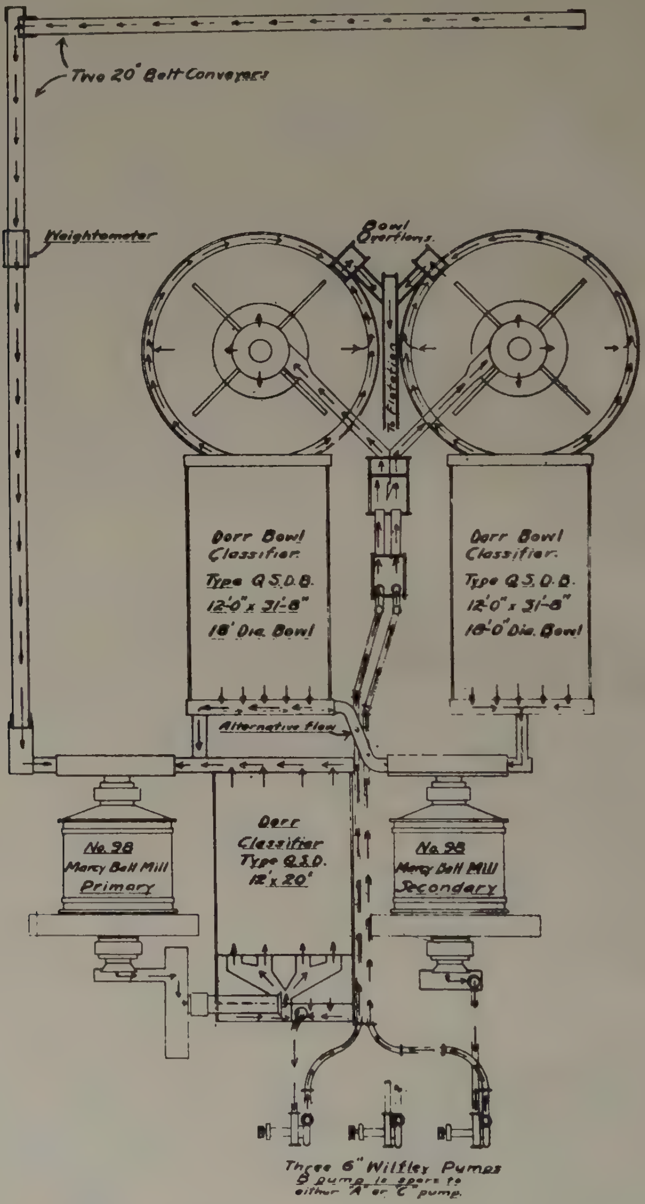
1. Symons primary ore bins, 3000 tons capacity.
2. Five 48-in. drum feeders.
3. Five 28-in. inclined belt conveyors, magnetic head pulleys.
4. Five 4 by 6-ft. type 60 Hum-mer screens, ½ by 1¼-in. mesh at 37° slope.
5. Five 5½-ft. Symons cone crushers—direct connected 150-hp. motors.
6. Main 60-in. collecting belt.
7. One 60-in. incline cross conveyor.
8. One 60-in. conveyor with automatic traveling tripper.
9. Symons secondary ore bins—4000 tons capacity.
10. Ten 30-in. drum feeders.
11. Ten 60 by 102-in. "Gyrex" screens—double deck—⅜ by ⅝-in. bottom screen.
12. Five 28-in. inclined feed conveyors.
13. Five short-head Symons crushers.
14. Five 24-in. belt conveyors.
15. One 42-in collecting belt.
16. One 42-in. cross conveyor.
17. One main 42-in. collecting conveyor.
18. One 42-in. inclined conveyor—No. 6.
19. One 42-in. conveyor with automatic traveling tripper.
20. Fine ore bins, capacity 6000 tons.

The original grinding circuit has undergone several changes, each, however, showing an improvement on the original. Diagrammatic flow sheets, Fig. 4, show the original plan and the present. At first primary and secondary grinding was employed. This proved very satisfactory from a tonnage standpoint, and the capacity was finally established at 1500 to 1600 tons per day per section at 86 per cent minus 200-mesh grinding. With this tonnage, however, the bowl compartments of the classifier were easily overloaded, resulting in a large amount of oversize in the overflow. To correct this condition the feed was introduced into the rake compartment through a manifold header pipe, the result being practically no load in the bowl. This allowed the speed of the bowl rakes to be reduced from 4 r.p.m. to 2 r.p.m. and the degree of fineness of the overflow increased from 86 per cent to 90 per cent minus 200 mesh. Some difficulty was experienced in balancing the load between the primary and secondary mills. It was decided, therefore, to try out single-stage grinding. This arrangement proved satisfactory and resulted in an increase of tonnage to 1000 tons per ball-mill day under normal conditions. The tonnage per mill is more or less regulated by the fineness of grinding, which is maintained at 90 per cent through 200 mesh.

Conversion of the secondary mills into primary mills was easily achieved. It was only necessary to install cross conveyors at the head end of the original feed conveyors and to furnish suitable splitter chutes to divide the feed between the two mills in each section. The primary 12-ft. four-rake primary classifiers have been divided at the center to make two separate machines. Gravity flow is obtained from the mill discharge to the classifier, and from the sand end of the classifier to the mill scoop box, by simply lowering the classifier 20 in. This at the same time does away with the original scoop lifter.

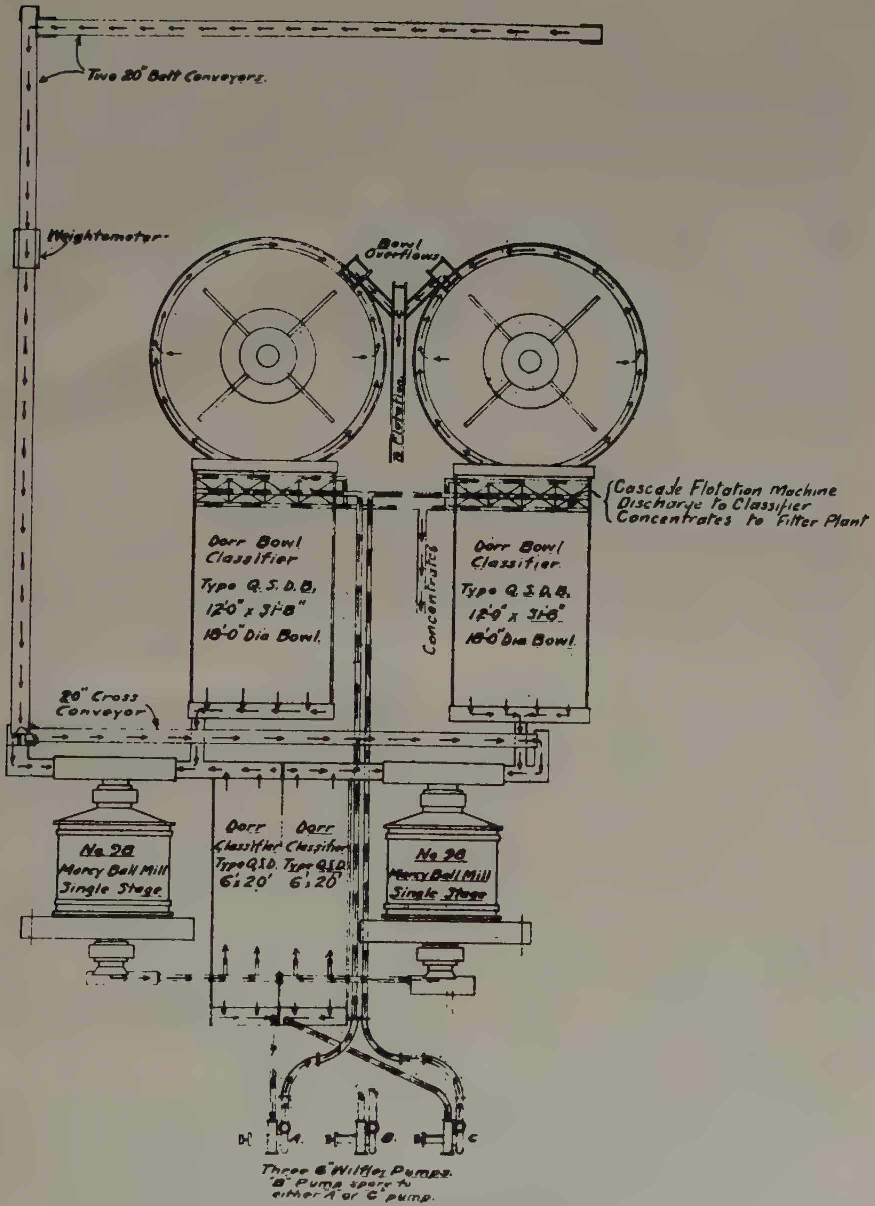
Increased tonnage from single-stage grinding can be attributed to two factors: (1) the method of feeding the bowl classifiers, which immediately showed a definite improvement in the amount of oversize carried in the overflow, making it possible to increase the tonnage with better classification; (2) the sticky nature of the primary slime furnishes the medium that causes a thick coating of pulp to adhere to the balls, which, of course, is ideal. This primary slime seems to have a lubricating quality that prevents wear to a certain extent. In ordinary two-stage grinding the absence of this slime in the secondary mills was decidedly noticeable in that the steel consumption was practically double as compared to the primary mills. This condition is not easily affected by the pulp density.

The mill site being practically flat, all return circuits are handled by Wilfley pumps. These pumps are all in duplicate and very few interruptions occur from pump failures. The original estimated life of pump wearing parts handling the relatively coarse primary classifier overflow was three weeks. This has been greatly exceeded and is now fairly well established at two months Pumps handling the finer prod-


Two 20" Belt Conveyors
Weightometer
Bowl Overflows
To Flotation
Dorr Bowl Classifier. Type Q.S.D.B. 12'-0" x 31'-8" 18' Dia. Bowl
Dorr Bowl Classifier. Type Q.S.D.B. 12'-0" x 31'-8" 18'-0" Dia. Bowl
Alternative flow
No. 98 Marcy Ball Mill Primary
Dorr Classifier Type Q.S.D. 12' x 20'
No. 98 Marcy Ball Mill Secondary
Three 6" Wilfley Pumps
B pump is spare to either A or C pump.

Unit as originally designed for two-stage grinding.

FIG. 4.—SHOWING CHANGES IN GRINDING FLOW-SHEET INCIDENT TO CONVERSION FROM TWO-STAGE TO SINGLE-STAGE GRINDING.



Unit as altered for single-stage grinding.

Fig. 4.—(*Continued.*)

ucts, such as flotation feed, concentrates, cleaner tails, etc., have a life of well over 2½ years.

The ball-mill bay is served by a 90-ton crane capable of lifting a fully loaded mill. A spare mill with a full charge of balls is kept in reserve; therefore it is only necessary to lift out a mill needing repairs and put in place the spare mill. Approximately 6 hr. is required to make this change. A mill undergoing repairs is placed in a rack fitted with roller trunnions, where the mill can be revolved at will and every part reached from the working platform. Relining requires 8 to 10 hours.

Cost distribution of grinding, classification and pumping per ton, on a basis of 8000 tons per day, follows: labor, 0.03s.; power, 0.30s.; grinding material, 0.23s.; maintenance, supplies, etc., 0.05s.; total: 0.61s.

Flotation

Flotation problems may be classed under three separate headings; grinding, oxides and alumina control. The first of these, having a definite effect on both grade of concentrates and recovery, is the most important. The degree of dissemination of the copper minerals and the softness of gangue are the controlling factors. Microscopic analyses show that practically 85 per cent of the sulfides lost in the tailings are in the form of chatty material, with grinding at 90 per cent minus 200 mesh. This is true in elutriator products as fine as 800 mesh. In the coarser sizes (over 250 mesh) 95 per cent of the visible sulfides is attached. The economic limit of the fineness of grinding is dependent upon the selling price of copper.

Oxides are of secondary importance at present, as this is only a temporary condition resulting from the mining program (see p. 937). Increased recoveries can be made by floating part of the oxides. The resulting concentrate, however, would be too low in grade to smelt at the present price of copper. Laboratory tests indicate that a saving of one to two pounds of copper per ton can be made by floating a portion of the oxides.

Control of alumina offers a rather difficult problem. Smelting costs increase in proportion to the amounts of alumina in the concentrate. The ideal concentrate for smelting should carry an excess of silica, but as the gangue (shale) is composed chiefly of silica and alumina, the differentiation is practically impossible. The logical alternative, therefore, is to produce a concentrate with as low an alumina content as possible, which means holding the grade at 56 to 60 per cent Cu. In making this grade it is necessary to sacrifice recovery to a certain extent, but under present conditions it is economical.

The following assays will give some idea of the average contents of the Roan concentrates in recent production: total Cu, 58.51 per cent;

oxide Cu, 0.59; SiO_2, 11.52; Al_2O_3, 4.08; Fe, 5.27; S, 17.78; CaO, 0.15; MgO, 0.57; residue, 4.12.

The flotation circuit consists of primary and secondary roughers and primary and secondary cleaners. The machines are the pneumatic matless cell type. The primary roughers are 22 ft. long and the secondaries 60 ft., while the cleaner cells are 22 and 16 ft. respectively. Feed is introduced at the head end of the primary roughers, which make a concentrate that goes to the cleaner circuit and a tailing that goes to the secondary roughers. These produce a secondary concentrate, which is returned to the head of the primary roughers, and a final tailing. The primary rougher concentrates enter the head end of the primary cleaner cells, which make a concentrate and tailing. The concentrates go to the secondary cleaner cells, which make a finished concentrate with tailings returned to primary cleaner. The primary cleaner tailings go to a regrind unit, which consists of a 35 by 6-ft. Dorr thickener with rakes speeded up to one revolution in 1½ min. with the underflow going to a 4½ by 12-ft. ball mill. The mill operates in closed circuit with the thickener, the overflow from which is treated in separate flotation cells. Concentrates are returned to the regular cleaning circuit and tails to waste. The product treated in this unit assays normally from 6 to 8 per cent copper, principally in the form of gangue-mineral attachments. Flotation results on this product were practically nil before regrinding, whereas when reground, say to 325 mesh, a 50 per cent concentrate with a 0.5 tailing is not above normal. The tails are higher or lower, depending on the amount of oxide present. In other words, the total tails assay is generally just double the oxide assay.

Flotation costs per ton are segregated as follows: labor, 0.02s.; reagents and royalties, 0.11s.; low-pressure air, 0.07s.; supplies, etc., 0.03s.; total, 0.23s.

Wet samples are taken automatically by Geary Jennings electric samplers. Shift samples are filtered and dried by flotation operators, while cutting down and further handling is done by a native helper under the supervision of the testing engineer. Moisture samples on mill feed are taken by the ball-mill operators and dried to constant weight in a specially built heat-controlled Westinghouse electric oven. Control check samples on feed, concentrates and tailings are taken over 24-hr. periods, and are handled by the testing engineer only.

The flotation reagents used are potassium ethyl xanthate, cresylic acid and sometimes pine oil. The amounts used are: xanthate, 0.060 lb. per ton; cresylic acid, 0.16 lb. per ton; pine oil, 0.001 lb. per ton.

Reagents are stored and handled in a separate building equipped with off-loading platform, overhead conveyor system and storage tanks. Mixing vats for chemical reagents are placed immediately above pressure tanks from which the solution is forced under air pressure to the mill reagent feeders through 1½-in. pipe lines. Oils are handled by the same means.

Railway service tracks enter the mill at the repair bay where the 90-ton crane is available for the off-loading of balls, liners and other supplies.

Finished concentrates at present are thickened in two 35 by 10-ft. Dorr thickeners. The overflow water is returned to the mill circuit by gravity, while the thickened concentrates at 70 per cent solids are pumped 1700 ft. to three stock tanks or direct to 8 by 8-ft. Oliver filters. Special Wilfley type C.A. heavy-duty 3-in. pumps are used for this work. The pumping of concentrates is a continuous operation and no difficulty is experienced with the pipe line choking in spite of the fact that the distance is 1700 feet.

The latest development in this department is the use of cascade flotation cells in the ball-mill circuit. These cells are placed directly above the bowl-classifier rake compartment with the tailings discharging direct into the classifier. The concentrate is of sufficiently high grade to go direct to the filter plant. Results to date indicate that it is possible to make a recovery of approximately 50 per cent of the total copper in the mill feed. The concentrate is much coarser than that produced on the air-lift machines. The chief value of this operation is that of preventing, to a large extent, the overgrinding of freed mineral, which under ordinary straight classification is difficult to prevent. It further reduces the load on the air-lift machines to such an extent that it is necessary to operate 80 per cent of these machines only, with a consequent saving of power.

Concentrate-handling costs are 0.01s. per ton equally divided between labor and electric power.

In addition to the three 8 by 8-ft. Oliver filters, the principal items of equipment in the concentrate filter plant are three 18 by 9-in. Worthington vacuum pumps, two 12 by 9-in. Worthington compressors, and three mechanically agitated stock tanks. A series of three 20-in. conveyors delivers the filtered concentrates to three stock bins, each equipped with belt feeders discharging to an inclined conveyor which, in turn, discharges into a specially built weigh hopper in which the product is weighed before going to the smelter stock bins.

At this point automatic samples are taken immediately before weighing and assayed for copper, moisture and gangue minerals. These weights and assays form the basis for the metallurgical balances for the mill and smelter, the mill organization being responsible for all copper on its side of the weigh hopper and the smelter for all copper delivered from the weigh hopper to the smelter bins.

The only outstanding feature of the filtering operation is the speed at which the filters are run. It was found that the filter cake had a tendency to build up too rapidly, and break away from the drum at the point where it leaves the pulp in the tank. Various dilutions were tried, but with slight success. The filter was then speeded up from one revolution in 4 min. to one in 1½ min. The thickness of the cake was reduced from 2½ in. to a maximum of 1 to 1¼ in. with no difference in the moisture content. Two filters operating a total of 48 hr. are now able to take care of the entire present mill production of 375 tons of concentrates per day.

Filtering costs are 0.01s. per ton, largely represented by operating labor.

TAILINGS

Tailings flow by gravity through 2500 ft. of 18-in. Victaulic steel pipe to a 250-ft. Dorr traction thickener. The clear-water overflow is returned to the mill circuit with sufficient mine water added to make up for losses. Mine water, which is slightly alkaline with a pH value of 8.1, is added to the thickener and serves to dilute the tailings so that settling is more rapid and a clear overflow is obtained.

The thickened tailings (55 per cent solids) are pumped to the tailings-dam pipe system by 6-in. high-pressure Wilfley pumps. There are four pumps, each capable of delivering 6000 tons per day through 12-in. steel pipe extending the length of the dam and the emergency dam, a total of 7000 ft. The uptake pipes are spaced at 800-ft. intervals on the main dam and at 500 ft. on the emergency dam.

Dam building is a comparatively simple matter after a toe wall is established. The original method of distributing tailings was by means of a wooden launder system extending the length of the dam and about 50 ft. inside the toe. Two-inch down pipes carried thickened tailings to the toe wall. The toe wall is kept up by natives, each with an allotted task of 20 yd. of wall 2 ft. high per day. This method of working calls for a minimum of supervision and at the same time is not a difficult task for the boys, as they are generally finished in 5 to 6 hr. This method of working native labor has proved very satisfactory, both from the native's point of view and the employer's, but can be used only on routine work of this kind. The use of tailings launders has been discontinued.

At present thickened tailings are pumped through a 12-in. pipe line at the foot of the dam with uptake pipes at intervals of 200 yd. Ditches dug along the top of the dam wall distribute the tailings to any point. The handling of tailings and dam building are greatly simplified, and at a cost of only one-tenth of the original.

To overcome the erosion of the dam wall caused by the heavy rainfall (50 to 60 in. per year), a system of thatching is being tried with apparent success. A layer of long-stemmed grass, such as that used in building native huts, is laid the length of the wall being built, the tops of the grass projecting outward about 2 ft. from the wall. The new wall is then built on this layer of grass, making a thatchlike covering, which practically stops all erosion. Serious difficulties were anticipated in building a dam of material as fine as 90 per cent minus 200 mesh, but the wall stands perfectly at about 30° from vertical.

Tailings-disposal costs amount to 0.03s. per ton milled, distributed between labor, power and supplies.

In general, with ores averaging 3.15 to 3.25 per cent total copper and 0.15 to 0.25 per cent oxide copper, the Roan Antelope concentrator will produce a concentrate of 58 per cent copper, representing 87 to 88 per cent total recovery and 90 to 91 per cent sulfide copper recovery, with a ratio of concentration of approximately 20 tons into one.

GENERAL COSTS

General costs on a basis of a tonnage of approximately 8000 tons per day are summarized by processes as follows:

PROCESSES	COST PER TON ORE MILLED, SHILLINGS
Crushing, conveying and screening	0.10
Grinding and classifying	0.61
Flotation	0.23
Concentrate handling	0.01
Filtering	0.02
Tailings disposal	0.03
Assaying and sampling	0.02
Mill water	0.01
General concentrator expenses	0.04
Total concentrating costs	1.07

RESEARCH WORK

A fully equipped research laboratory is maintained, where routine testing is done on ores from new mine headings as well as investigations on new reagents or methods tending to improve mill efficiency. In order to prove up on laboratory work a full-scale test unit is now under construction. It will be possible to separate this unit from the remainder of the mill, thereby avoiding the danger of upsetting the metallurgy of the entire mill when making tests. The flow sheet will consist of a 9 by 8 Marcy ball mill operating in closed circuit with a Dorr bowl classifier for the grinding unit. The flotation section follows the general plan of the mill, space being left for new developments in flotation apparatus. There is also in this unit a regrind ball mill and classifier for regrinding intermediate products such as cleaner tails and secondary rougher concentrates. A 50-ft. tailings thickener is provided to keep the return water circuit separate from the mill. Final concentrates will be filtered with the regular mill product.

Suggestions from any of the employees or others tending to improve metallurgical results or working conditions are acted upon promptly. In this we are particularly fortunate in having the whole-hearted backing of the General Manager, who not only furnishes the incentive for new ideas, but the facilities for thoroughly trying them out.

Concentration of Polish Bleischarley Ores

By L. P. Davidson* and M. C. Messner,† Members A.I.M.E.

The Giesche Spółka Akcyjna, in Polish Upper Silesia, produces zinc, lead and coal, together with many byproducts emanating from the zinc-lead ores. The development of the concern in the 230 years of its existence has been influenced not only by metallurgical improvements but also to an unusual extent by political and economic factors.

The company was founded by George Giesche, who was born in 1653 and died in 1716, and who in 1704 received from Emperor Leopold I the exclusive privilege of mining and selling *galmei*[1] in Silesia. His children, who retained the business, gave the name to the firm—George von Giesches Erben.

After 1802 modifications in the Prussian mining and corporation laws limited the rights formerly enjoyed. The family concern became a corporation and expanded its activities, building smelters and opening new mines. The establishment of the present eastern boundary of Germany resulted in the transfer of nationality of a large part of the mines and surface plants to Poland, which, in turn, necessitated the formation of a Polish company, the Giesche Spółka Akcyjna.

The first concentrators at the Bleischarley mine were built between 1854 and 1860 and owing to dual ownership were two in number. They were used only for the treatment of galena. In 1873 a concentrator was built for the treatment of sulfide ore preparatory to treatment in the Recke roasting plant, which was the first to have an acid plant for recovery of the sulfur gas.

This concentrator was replaced by a larger one in 1888, when the present orebody was opened up. At the same time, a mill was built for the treatment of the galmei. The concentration of the sulfide ore resulted in a much higher grade of feed for the zinc plants after the first troublesome steps of the roasting process had been worked out. Consequently, the oxide ore in its primitively concentrated state was no longer highly valued as a retort charge and steps had to be taken to increase its zinc content.

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[1] The German word for oxidized zinc ores, which are partly carbonate, partly silicate, and usually found with a dolomitic gangue.

These mills were worked chiefly by women. Hand sorting and jigging were the only methods used to separate the mineral from the gangue. No provision was made for treating slimes, and they were settled in ponds and stockpiled. These old slime dumps are a valuable source of zinc today.

Improved social legislation and technology made modernization of plant and practice imperative, and in 1910–1911 the new concentrator was built at the Bleischarley mine. A full description of this concentrator was published in *Glückauf* in 1912, therefore a detailed description is not given here.

The interdependence between the mining and milling operations due to the absence of storage facilities other than the ore contained in the hoisting cars gave rise to operating troubles both in the concentrator and the mine. The adoption of the 8-hr. day in 1918 gave rise to other changes, so that the plans of 1911 became somewhat antiquated by 1926, and economic and political considerations dictated a change in metallurgical practice.

The general plan for the new process was worked out by Messrs. Frederick Laist, General Metallurgical Manager of the Anaconda Copper Mining Co., George Sage Brooks, President of the Giesche Spółka Akcyjna and Ernest Klepetko, then Metallurgical Manager of the Giesche Spółka.

Experimental tests had shown that the wurtzite, the chief sulfide mineral of the orebody, was easily amenable to flotation. Working from this point, flotation was decided upon for the slime and the low-zinc jig middlings. By installing belt elevators and changing the gravity flow sheet to some extent, the production of gravity concentrates of higher grade was made possible.

Further experience with the operation permitted simplifications in the flow sheet originally planned, so that at the present time the concentrator at the Polish Bleischarley mine produces flotation concentrates assaying 61 per cent zinc, one-half of which comes from the primary flotation of slime and jig middlings, the other half from the flotation of iron out of the gravity concentrates, the tailings from this operation being the finished zinc concentrate. The operation is conducted by Polish workmen, supervised by Polish engineers and is not dependent on import for any supplies, reagents or repair parts. It is an example of nationalistic self-sufficiency.

The Modern Mine and Mill

The Bleischarley zinc-lead mine is situated in Polish Upper Silesia, 9 miles north of Katowice, Poland, and 2 miles east of Beuthen, Germany, the international boundary forming the western boundary of the property.

The ore occurs as zinc blende (wurtzite) and galena in the ratio of about 10 Zn to 1 Pb, or a mixture of these sulfides with their oxidation products, replacing dolomite or filling cavities and fractures therein. Marcasite is present in varying amounts.

The principal hoisting unit is Kraker shaft, a four-compartment shaft equipped with two similar electric hoists located at the top of the headframe and vertically over the shaft. The cages are in balance and each carries two mine cars having a capacity of 0.35 cu. m. which are transported to a tipple that empties them into a bin that will hold 250 cu. m. The empty cars roll by gravity back to the cage.

The storage bin is of reinforced concrete construction and is provided with two discharge openings. Under these openings are two pan conveyors which serve as the bottom of the bin and convey the ore to the 100-mm. grizzlies installed ahead of the Blake crushers. They discharge the oversize directly to the crusher, the undersize joining the crushed ore on a conveyor belt.

The crushed ore together with the grizzly undersize is conveyed by three conveyor belts in tandem to the feed bins over each of three of the sections in the gravity mill. The first belt has a slope of 12° and is 119 m. long, the second is 112 m. long and has the same slope, and the third belt is horizontal and 185 m. long. The belts are rubber-covered six-ply canvas and can carry 120 metric tons per hour.

The belts do not discharge directly on to the succeeding belt but on to a revolving steel drum. This drum has reduced the wear and tear on the belts immensely, both by eliminating the direct fall of the sharp rock on to the rubber surface and also because the skirt-boards at the discharge point can be set farther away from the belt.

All the incoming ore is weighed automatically on the first belt and is sampled as it falls from the second belt to the third. It is removed by a traveling tripper to intermediate bins over each of the three sections in the mill devoted to the concentration of sulfide ores. Each section will treat about 300 tons of ore in an 8-hr. shift.

Feeding, Screening and Classifying

A reciprocating pan feeder delivers the ore from the section storage bins to a conical trommel with 45-mm. openings. The oversize goes to a sorting table and the undersize to a train of trommels of various sizes. From these trommels all products go to jigs. The undersize from the 1.5-mm. screens passes over hydraulic classifiers, from which the spigot goes to jigs and the overflow to the slime-settlement tanks. A list of the trommels for one section is given in Table 1.

Fig. 1 shows diagrammatically the arrangement of the trommels, jigs, rolls and classifiers in one section. The 3.5-mm. trommels are in duplicate, as well as the 2.5-mm. and the 1.5-mm. The sizing for the sand jigs 1, 2 and 3 is effected by the hydraulic classifiers.

TABLE 1.—*Trommels for One Section*

Opening, Mm.	Number of Trommels	Dimensions, Meters		
		Length	Diameters	
45.0	1	3.0	1.1	1.4
28.0	2	3.0	1.0	1.3
20.0	1	3.85	1.1	1.7
14.0	1	2.9	0.9	1.2
10.0	1	2.9	0.9	1.2
7.0	1	2.9	0.9	1.2
5.0	1	2.9	0.9	1.2
3.5	3	2.9	0.9	1.2
2.5	2	2.9	0.9	1.2
1.5	2	2.9	0.9	1.2

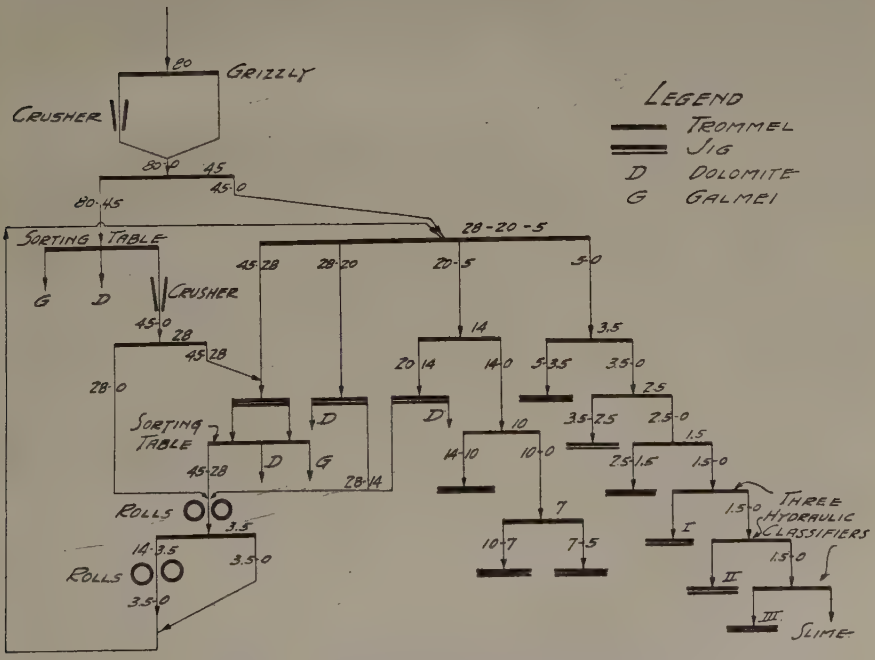

FIG. 1.—FLOW SHEET OF GRAVITY SYSTEM.

The ore-dressing practice of 1910 made every possible provision for desliming the ore and for the production of as little secondary slime as possible. It has been the aim of the present operating staff to utilize the existing equipment in so far as it is metallurgically and economically feasible, therefore the maintenance of the complicated screening and classifying system appears somewhat anachronistic, but under the existing conditions is completely justifiable.

HAND SORTING

The sorting is carried out on two sizes of material; namely, 80 to 45 mm. and 45 to 28 mm. The trommels deliver a washed feed to the sorting tables, so that the separation of the sulfides, barren dolomite and galmei is easy. The first sorting table is 5 m. in diameter and makes one revolution in 1.75 min. The feed is the oversize from the 45-mm. trommel. About 12 per cent of the weight of ore is discarded from these tables as finished tailing. Such galmei as is free from sulfide minerals is sorted out and stocked separately. The material left on the table goes to a 500 by 260-mm. crusher set at 45-mm. opening. The crushed product is screened on a trommel with 28-mm. openings, the undersize joining the middlings from the second sorting table, the oversize going to a 45-mm. jig.

The product from the jig goes to a second sorting table 7 m. in diameter and making one revolution in 3 min. Both the concentrates and tailings are sorted, some galmei being removed from both the tailings and the concentrates.

The tailings from the sorting tables go to the waste dump. They comprise about 20 per cent of the ore fed to the mill and assay about 0.9 per cent Zn, about half of which is in the form of carbonate or silicate, which permeates the dolomitic gangue. These tailings are stocked separately and during the building season are sold for road ballast and concrete work.

The middlings from the tables are crushed through rolls set to 10 mm. and screened through a 3.5-mm. screen. The oversize is crushed in a set of Allis-Chalmers rolls set to 3.5 mm. and joins the undersize from the first trommel. The crushed middlings are elevated back to the head of of the trommel system for screening and jigging.

JIGGING

The jigging is done with materials of the following sizes: 45 to 28 mm., 28 to 20 mm., 20 to 14 mm., 14 to 10 mm., 10 to 7 mm., 7 to 5 mm., 5 to 3.5 mm., 3.5 to 2.5 mm., 2.5 to 1.5 mm., and three graded sizes under 1.5 mm. on products from the hydraulic classifiers. As mentioned above, both tailings and middlings from the 45 to 28 jigs go to the sorting tables. Finished tailings and middlings are made on the 28 to 20 and 20 to 14-mm. jigs, the middlings being crushed in the Allis-Chalmers rolls and returned to the head of the trommel system.

The next three sizes, 14 to 10, 10 to 7, 7 to 5 mm. produce lead concentrates, middlings and finished tailings. The middlings are returned with the other middlings to the head of the trommel system after crushing to 3.5 mm.

Gravity zinc concentrates are produced first from the 5-mm. jigs, which are of the five-compartment type with compartments measuring 820 by 580 mm. The products and their destination are in general as shown in Table 2. A single section has the jigs listed in Table 3.

TABLE 2.—*Jig Products*

COMPARTMENT	PRODUCT	DESTINATION
1	Lead concentrate	Lead smelter
2	Marcasite and galena	Re-treatment for galena
3	Zinc blende and marcasite	Flotation, system 3
4	Zinc blende	Flotation, system 3, or shipment
5	Zinc middlings	Flotation, system 1

TABLE 3.—*Jigs of a Single Section*

Size of Feed, Mm.	Compartments	Number of Jigs	Pulsations per Minute	Type of Jig
45–28		1	90	Pipe discharge
28–20	3	1	110	Pipe discharge
20–14	3	1	130	Pipe discharge
14–10	5	1	140	Pipe discharge
10–7	5	2	160	Pipe discharge
7–5	5	2	180	Hutch
5–3.5	5	2	210	Hutch
3.5–2.5	5	3	240	Hutch
2.5–1.5	5	2	260	Hutch
Sand 1	5	2	280	Hutch
Sand 2	5	1	280	Hutch
Sand 3	5	1	300	Hutch

All jigs, sorting tables, rolls, trommels and feeders are driven from a line shaft, one 200-hp. motor driving two sections.

The marcasite product from the second compartments of the jigs is at times combined with the galena and shipped to the lead smelter. At other times, when the lead plant does not want so much iron, this product is rejigged and a better separation of the galena and marcasite is made, the marcasite being stocked for use in acid plants.

FLOTATION OPERATION

The construction plan of 1927–1928 foresaw the treatment of the slimes in one system of the flotation plant and provided for two systems for the treatment of middlings from the gravity mill.

Two separate and distinct flotation circuits are in use. One system treats the gravity concentrates produced, in which the iron is floated out. Obviously, the treatment of such a material results in an iron concentrate containing too much zinc to be wasted. This iron concentrate is combined with the slimes produced in the gravity mill, thickened and returned to the other flotation system, where the middlings from the gravity mill are treated. In this system the iron is depressed and is discarded with the dolomitic tailing.

Intensified rejection of gravity tailings took such a load off the so-called sand sections that the equipment could be utilized to produce all of the zinc concentrate as flotation concentrate. The former slime section was used for the re-treatment of the gravity concentrate and the two sand sections were combined in tandem for the treatment of the thickened slime, the iron-zinc product from system 3 and the gravity middlings. In the first section a high-grade rougher concentrate was made while in the second no effort was made to attain a good grade of concentrate but solely to obtain a low tailing. Fig. 2 shows the flow sheet of the flotation systems.

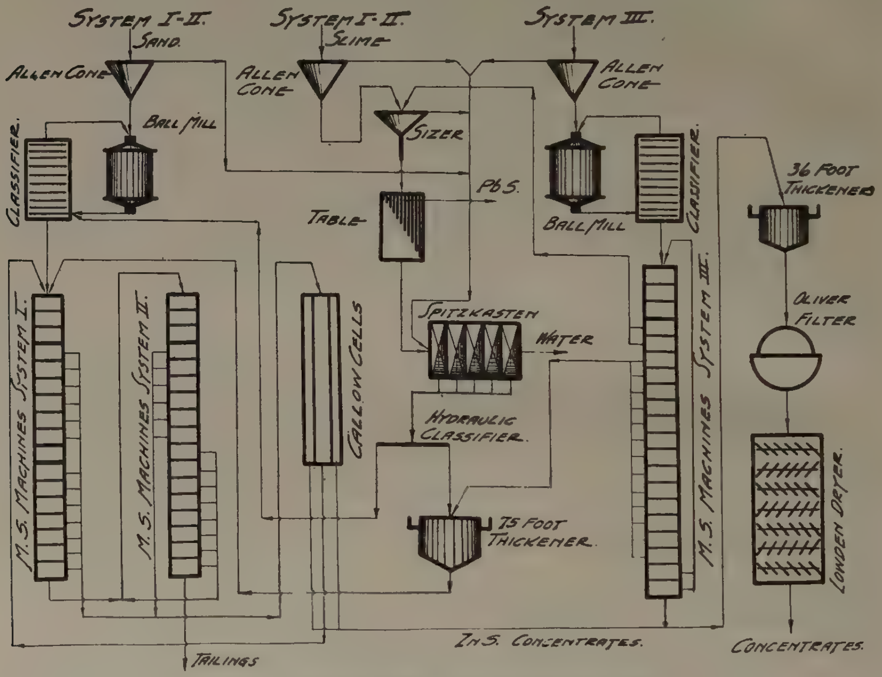

FIG. 2.—FLOW SHEET OF FLOTATION SYSTEM.

The jig middlings are dewatered in Allen cones 4.5 ft. in diameter, which discharge into steel feed bins. The Allen cone overflow combines with the other slime and is clarified in settling basins 3 by 39 m. in area.

The coarse material is fed from the bins to the ball mills by a reciprocating push conveyor. This is a standard push conveyor originally designed for the conveying of hot calcine, but it has worked very well on the wet ball-mill feed.

The slimes from the hydraulic classifiers in the gravity mill, together with the concentrate from the first two cells of the machines in system 3, pass through Allen cones and a Fahrenwald classifier. A small amount of galena is recovered on a table, the tailings from which combine with the overflow of the cones and classifier in the settling basin mentioned above. The thickened product from the settling basins is classified in a hydraulic classifier, the sand from which goes to the Dorr classifier of the sand mill. The purpose of the classification is twofold: (1) for better operation of the 75-ft. thickener and (2) to reduce the ball-mill feed by sending only the sandy portion of the slimes to the classifiers, the remainder going directly to the flotation machines.

The purpose of tabling the underflow from the Fahrenwald classifier is to reduce the lead content of the flotation concentrate. This operation is carried out only because the equipment was available and because a slight decrease in the lead content of the zinc concentrates makes for easier operation in the smelting plants.

Fine Grinding and Classifying

The ball mill and classifier are of standard design, the mill discharging into the classifier sump and the classifier sands returning to the scoop feeder of the ball mill.

There are four cylindrical ball mills, three being 2 by 2.5 m. and the fourth 2 by 2 m. The liner plates for the ends are bolted through the shell. The side liners are manganese-steel rods held in place by the end plates and bolted through the shell only at the manhole. The large mills are driven by 150-hp. motors, the small mill by a 100-hp. motor, at 20.5 r.p.m.

The ball load is approximately 12.5 metric tons of 3-in. balls, also of manganese steel.

The classifiers are standard Dorr D duplex classifiers 6.4 m. long by 1.84 m. wide, and make 20 strokes per minute. They are set on a 16° slope.

At the present, nine flotation machines out of the fourteen installed are in operation. The machines are standard M.S. subaeration type, the machines in systems 1 and 2 having 17 cells and in system 3 having 20 cells. The cells are 30 in. square by 36 in. deep. The impellers are 18 in. in diameter and make 338 r.p.m. They are shrouded both top and bottom. The bottoms of the cells and the sides are protected against wear by cast-iron liners. Each machine is driven by a 68-hp., 725-r.p.m. motor with a belt drive.

The agitating cells are equipped with cruciform impellers, the blades of which are set at 45° to the vertical. Cross wooden baffles are mounted in each conditioning cell.

The machines in system 1 are provided with double skimmers, at the front and the back of the machine. This was done to take as much load as possible off the machines in system 2, where the final tailings are made. The feed to these machines averages 10 metric tons per hour with a zinc content of about 20 per cent.

The rougher concentrates are cleaned on four five-pan Callow cells situated between systems 2 and 3. The coarser portions of the rougher concentrates are high in zinc, so the feed to the Callows passes through a hydraulic classifier, the coarse concentrate bypassing the cells and combining with the cleaner concentrate. The cleaner tailing is returned to the head of system 1.

Thirteen cells from the machines in system 1 make a rougher concentrate, the other four cells being used as conditioning and mixing cells. Six cells of the machines in system 2 make a rougher concentrate, the following eight cells making a middling which returns to the head of the machine.

The flotation of the gravity concentrates is very simple. Two of the ball mills are used for grinding the gravity concentrates. Only two flotation machines are in operation, treating about 7 tons per machine hour. There are three conditioning cells followed by two cells from which the product goes to the Fahrenwald classifier. Thirteen cells deliver a product that goes to the 75-ft. thickener and the last two cells make a middling that is returned to the head of the machine. The "tailings" go to the concentrate thickeners together with the Callow concentrates.

REAGENTS

The reagents in use at the present time are burned lime, copper sulfate, sodium xanthate, wood tar oil and steam-distilled pine oil. All reagents are prepared in a separate building adjoining the mill and are either pumped or trammed into the flotation mill. Lime is purchased in lump form, crushed through a Blake crusher and a set of rolls to approximately 1 mm. This is then trammed to the mill and fed by a feeder actuated by the ore feeder for each mill. The lime is dropped into a mechanically agitated tank filled with hot water. The overflow from the tank goes to a small settler, the overflow from which goes to the flotation machines in system 1 and to the slime settlers. The latter is to insure a clear overflow from the 75-ft. thickener and a suitable alkalinity in the thickened slime.

Copper sulfate is dissolved and pumped to a tank in the mill from which it is drawn to the individual feeders. These feeders are brass disks carrying copper cups. The regulation is obtained by varying the number of cups on the disk. Lead pipes convey the solution to the flotation feed launders.

Oils and xanthate are also pumped into intermediate tanks in the mill, one to each system. They are fed by pulley feeders of usual design. The distributors are revolving with removable wells, so that any number of machines from one to four may be served at one time.

DEWATERING FLOTATION CONCENTRATE

The flotation concentrates are thickened in Dorr thickeners 36 ft. in diameter by 8 ft. 4 in. deep. They have a conical false bottom of concrete. The concentrates are pumped by Dorrco duplex pressure pumps to the Oliver filters 52 m. away. Three thickeners are available but generally only two are in use, being operated in tandem.

There are four 5 by 8-ft. Oliver filters, of standard design, which deliver the filtered concentrates to Lowden driers. The filter cake is discharged with about 10 per cent moisture. The severity of the winters and the use of a part of the concentrates for the production of acid in chamber plants made it desirable to dry the concentrates before shipment, therefore the Lowden driers were installed. They are 48 ft. long by 12 ft. wide and are housed in a room with the filters, adjacent to the flotation mill.

Each drier has the driving mechanism at the discharge end, the firebox being directly under the filters at the feed end. The plow mechanism is supported from four crossbeams, which in turn are supported at each end by concrete piers. These piers must be far enough from the sides of the drier so that they are not affected by expansion.

The dried concentrates are discharged to a conveyor belt, which carries the product to storage bins. The concentrates are shipped to the smelters in special closed cars or are loaded into standard ore cars if the shipment is made over the standard-gage railway.

METALLURGICAL DATA

Table 4 shows operating data for one typical month. The oxide zinc in the feed was 1.74 per cent, or 9.1 per cent of the total. As only 0.54 per cent was recovered in the galmei concentrate, the remainder of the oxide zinc went to the tailings. This indicated a sulfide recovery of slightly over 95 per cent with an iron rejection of 91.6 per cent.

With low metal prices obtaining, it was desirable to sacrifice some recovery to maintain low operating costs. Up to the summer of 1931, the loss in jig tailings was about half of the amount shown in the table. By increasing the amount of jig tailing produced at the sacrifice of one per cent in recovery, the amount of feed to the flotation system was cut by about 10 per cent. This was reflected in the costs of flotation reagents and fine grinding and more than offset the loss in metal.

The grinding is fine for zinc ore. The screen analysis shows 10 per cent on a 100-mesh screen and 73 per cent through 200 mesh. The fine grinding is necessary for recovery, probably because of fine crystals of blende intergrown with dolomite or marcasite, or both together.

The flotation of the slime and sand middlings is carried out in an alkaline circuit, a pH of 10.8 being the optimum alkalinity. A part of the lime is added as milk of lime to the slime before thickening. The lime that settles out during the slaking is sent to the ball mill, the rest being fed to the elevator boot ahead of the flotation machines. The other reagents are added as follows: copper sulfate, 60 per cent to the head of the flotation machines in system 1, and 30 to 40 per cent to the middle cell of the machines, the rest being fed to the head cell in the machines in system 2; pine oil and wood tar oil are added 50 per cent to the head of the machines in system 1, 20 per cent to the middle cells and the remainder to the head cells in system 2; sodium xanthate is added at four points, 25 per cent at the head of system 1, 25 per cent to the middle cells in the same system, 10 per cent at the head of system 2, and the remainder at the middle cells of system 2.

TABLE 4.—*Operating Data for a Typical Month*

	Tons	Assays, Per Cent			Percentage of Total		
		Zn	Pb	Fe	Zn	Pb	Weight
Feed	19,583	19.04	2.80	5.99	100.00	100.00	100.00
Zinc concentrates flotation	5,397	60.80	1.51	1.82	88.08	14.88	27.56
Zinc concentrates galmei	160	12.51	0.77	6.88	0.54	0.22	0.82
Lead concentrates	555	4.53	71.43	4.81	0.67	72.24	2.83
Pyrite tailing	17	8.74	4.03	36.20	0.04	0.13	0.08
Hand-sorted tailing	4,051	0.78	0.06	3.37	0.86	0.46	20.69
Jig tailing	4,104	2.09	0.11	4.38	2.30	0.85	20.96
Flotation tailing	5,300	5.28	1.16	12.89	7.51	11.22	27.06
Total tailing	13,472	2.96	0.51	7.46	10.71	12.56	68.79

TABLE 5.—*Reagent Consumption*
Kilos per Ton Flotation Feed

Reagent	Systems 1 and 2	System 3
Lime	10.86	0.07
Pine oil	0.08	0.24
Copper sulfate	1.86	
Wood tar oil	0.06	
Sulfuric acid		0.02
Sodium xanthate	0.58	0.23

In system 3, the flotation is carried out with a pH of 7.2, either acid or lime being added to maintain this pH. All the reagents in this system are added at the head of the machines. The reagent consumption in the month covered by Table 4 was as shown in Table 5.

Sodium xanthate is produced in an auxiliary plant situated at some distance from the mill, on account of fire hazard. Caustic soda solution is purchased at the Solvay plant near Krakow and shipped in tank cars. The solution is discharged to a storage tank, from which it is pumped by air pressure to the mixing tank. Proper amounts of carbon bisulfide and denatured alcohol are added and the agitation is carried out for a specified time. The mixture is diluted and stored in drums. This solution does not keep well and must be used within a week. Recrystallizing gives a stable salt, which will keep many months in the absence of air.

The power is purchased. The consumption for all purposes is about 31 kilowatt-hours per ton of ore treated. This includes the tramming of concentrates and tailings, repumping of water, lighting and the operation of vacuum pumps and air compressors.

For the treatment of 25,000 tons of wet ore monthly, an average of 263 people are employed, distributed as follows:

EMPLOYEES		SHIFTS	PER CENT
Superintendence, clerks	17	Operating	72.1
Laborers	156	Loading, tramming	9.9
Women	90	Track work	11.0
		Repairs	7.0

With the treatment of 25,000 tons monthly, the gravity plant is in operation only one shift per day with two or three sections running. The flotation plant operates on two shifts, the filters and driers on three shifts. No work is done on Sundays or holidays.

ACKNOWLEDGMENTS

In conclusion, the authors wish to thank Messrs. Frederick Laist and George Sage Brooks for permission to publish this paper, and Messrs. R. E. Thomas, Assistant Chief Engineer, and W. Stępiński, Superintendent of the Bleischarley concentrator, for their assistance in its preparation.

INDEX

Note: In this index the names of authors of papers and discussions and of men referred to are printed in SMALL CAPITALS, and the titles of papers in *italics*.)

A

- Abbe. *See* Microscope.
- Abrasives: mechanical preparation, 787
- ALLEN, A. W.: *Economical Techniques in Treatment of Gold Ore*, 538
- Alumina: fused: mechanical preparation, 778
- Amalgamation: barrel process in Black Hills, 604
  - economical techniques, 538
  - experiments with bromine and chlorine, 604
  - gold ore: cyaniding before or after, respective merits, 538
  - Homestake mills, 603
  - Montezuma-Apex Co., 565
  - Pioneer Gold Mines of B. C. Ltd., 678
- Amber: mechanical preparation, 789
  - production, 789
- American Institute of Mining and Metallurgical Engineers: officers and directors, 4
- American Metal Co. of Texas: Presidio mill. *See* Presidio.
- ANABLE, A. AND DORR, J. V. N.: *Fine Grinding and Classification*, 161
- Anaconda Copper Mining Co.: production of high-grade concentrate at Anaconda, laboratory investigation, 413
- ANDERSON, C. O., ILLIDGE, R. E., HOWES, W., HARBAUGH, M. D. AND BURRIS, S. J. JR.: *Milling Practice in Tri-State Zinc-lead Mining District*, 861
- Arsenopyrite: powdered: magnetic properties: effect of heat treatment, 523
- Asbestos: mechanical preparation, 790
  - Quebec, average yield, 791
  - Vermont, yield, 791

B

- Ball milling (*See also* Crushing and Grinding, Tube Milling):
  - ball size, rationing, 47, 76
  - circulating load: and classification, 54, 76
    - high, effect on work, 167
    - useful, calculating, 60, 66, 77
  - closed-circuit: trap for gold, hydraulic, 560, 568
  - critical speed, 26, 75
  - cylindrical mills, wet grinding: fundamental principles, 24
  - dead load, determining, 29, 77
  - feed rate, 53
  - fundamental principles, 24
- Ball milling: grindability of heterogeneous ores, 58
  - large vs. small mills, 51
  - new philosophy—power an index of mill operation and investigation from standpoint of variables, 24
  - power: an index of mill operation, 24
    - formula for estimating, 39, 77
  - pulp density, 52
  - slippage: required conditions, 95
    - tests, 33
  - stage grinding, 59
  - variables, set and induced, 43
    - as basis for investigation, 24
  - variables that affect performance, 94
  - Western Australia and New Zealand, 75
- Ball mills (*See also* Grinding Mills):
  - bearings, improving, advantages, 79
  - commercial, regulation, 67
  - efficiency, 94
  - formula for calculating diameter from capacity and length, 98
  - friction: comparison in small and large mills, 79
  - friction losses, estimates from weight and speed, 81
  - grinding efficiency improved by increase in diameter of mill, 86
  - larger size, advantages, 79
  - most efficient rotary mill, 165
  - power: analysis, 84
  - speed: formula, 94
  - weight, relation to net power required, 80
- Banket ore, 760
- Barite: mechanical preparation, 792
  - sources in U.S., 791
  - uses, 792
- BARSKY, G.: *Discussion on Principles of Flotation*, 236
- BARSKY, G., SWAINSON, S. J. AND HEDLEY, N.: *Dissolution of Gold and Silver in Cyanide Solutions*, 660
- BATES, B. R.: *Discussion on Ball Milling*, 74
- Bauxite: mechanical preparation, 793
- Beattie Gold Mines Ltd.: flotation-cyanidation mill, 690
  - milling costs, 696, 700, 701
- Bendelari diaphragm jig, 878
- Bentonite: mechanical preparation, 793
- BINSACCA, W. A.: *Milling Methods at the Concentrator of the Fresnillo Company*, 751
- Bird Dog mill: flow sheet, 873

Black Donald mine, Ontario: graphite preparation, 807
Black Hills (*See also* Homestake): history of gold, 597
  metallurgy: brief bibliography, 616
    review with particular reference to Homestake ores, 597
  pyritic smelting, 605
  Thoen Stone, 597
Black sands source of ilmenite, monazite and zircon, 815
Blanket strakes for recovery of free gold, 561, 567, 577
Blanket tables: Black Hills, 601
  self-washing, 562
  Witwatersrand, 764, 775
Bleischarley mine: zinc-lead concentration, 952
Blue earth: mining for amber, 789
Bond, F. C. and Maxson, W. L.: *Crushing and Grinding Characteristics as Determined from Screen Analyses*, 146
Bond, F. C., Maxson, W. L. and Cadena, F.: *Grindability of Various Ores*, 130
Borax: mechanical preparation, 817
Bradford breaker vs. grinding mills, 74
Bradley leaching process, 831
Broken Hill mining companies, investigation of principles of flotation, 189, 245, 267
Bryan, R. R. and Kuryla, M. H.: *Milling and Cyanidation at Pachuca*, 722
Bubble machine. *See* Flotation.
Buchans mill: climate affects construction, 859
  copper concentrate, from lead-zinc ore, 852
  costs, 860
  crew, 858
  crushing, 844
  de-coppering lead concentrate, 851
  filtering and drying, 852
  flotation practice, 848
  gold and silver in concentrates, 850, 851, 852, 853
  grinding and classification, 846
  lead concentrate, 852, 853, 855
  lead-iron concentrate, 853
  lead-zinc ore, assays, 848
  zinc concentrate, 853, 855
Buchans Mining Co. Ltd., 842
Burma Corporation, Ltd., investigation of principles of flotation, 189, 245, 267
Burris, S. J. Jr., Anderson, C. O., Illidge, R. E., Howes, W. and Harbaugh, M. D.: *Milling Practice in Tri-State Zinc-lead Mining District*, 861
Butte copper ores. *See* Anaconda.
Buxton Mining Co.: amalgamation experiment in Black Hills, 604

C

Cadena, F., Bond, F. C. and Maxson, W. L.: *Grindability of Various Ores*, 130
Caledonia mill, design, 600
Campbell, A. B., Coghill, W. H., Gow, A. M. and Guggenheim, M.: *Ball Milling*, 24
Canada: Quebec: asbestos yield, 791
Cananea Consolidated Copper Co.: grinding flow sheet, 170
Celo mines: kyanite preparation, 809
Cement: mechanical preparation, 793
Century Zinc Co.: flow sheet, Scott mine, 870
Chalcopyrite: powdered: magnetic properties: effect of heat treatment, 523
Chalk: mechanical preparation, 794
Chats. *See* Tri-State District.
Christmann, L. J.: *Discussion on Principles of Flotation*, 237
Clark, A. J.: leaching Homestake ore, 541
  Review of Black Hills Metallurgy, with Particular Reference to Homestake Ores, 597
Classification: Beattie Gold Mines, 697
  Black Hills, 607, 609, 614
  Buchans mill, 847
  Coniaurum mill, 619
  Dome mines, 621
  Fresnillo, 738, 751
  gold ore, Montezuma-Apex, 564
  Hollinger Gold Mines, 627
  Kirkland Lake, 652
  McIntyre Porcupine, 633
  Nkana concentrator, 928
  Noranda, 585
  Pachuca, 723
  phosphate rock, 473
  Pioneer Gold Mines, 680
  Polish Bleischarley ore, 954, 959
  Presidio mill, 712
  Roan Antelope, 939
  sand tailing: Homestake mills, 609
  Stokes law, 161
  Tri-State district, 886
  tungsten ore, 836
  Witwatersrand, 764, 768, 772
Classifiers: combining principles of thickener and cone, 585
  hydraulic vs. mechanical, 164
  Job cone, 772
  MacLean, 772
  mechanical: theory, 161
  mechanical vs. hydraulic, 164
  types, 162
Clays: mechanical preparation, 795
Clemmer, J. B. and Coghill, W. H.: *Soap Flotation of the Nonsulfides*, 449
Coghill, W. H.: *Discussions: on Ball Milling* 74, 78
    on Relative Floatability of Silicate Minerals, 505
    on Slime-coatings in Flotation, 410, 411, 412
Coghill, W. H. and Clemmer, J. B.: *Soap Flotation of the Nonsulfides*, 449
Coghill, W. H., DeVaney, F. D. and O'Meara, R. G.: *Advantage of Ball (Rod) Mills of Larger Diameters and Advantage of Improving Bearings*, 79
Coghill, W. H., Gow, A. M., Guggenheim, M. and Campbell, M.: *Ball Milling*, 24
Colloidal chemistry of pulp thickening, 178
Colloids: classes, definition, 179
Collophane. *See* Phosphate Rock.

- Compañia de Real del Monte y Pachuca. *See* Pachuca District.
- Concentration. *See* names of minerals and processes.
- Coniaurum Mines, Ltd.: gold ore concentration, 617
  - milling costs, 620
- Consolidated Mining & Smelting Co., Trail, B C.: fluorspar preparation at Rock Candy mill, 803
- Conveying: phosphate rock, 475
  - Presidio mill, 719
- Cook, R.: magnetic separation, 509
- Cooley jig, 878
- Copper: recovery from lead-zinc ore at Buchans, 852, 853
  - recovery with gold at Noranda, 578
- Copper concentrate: high-grade from Butte copper ores: laboratory investigations, 413
  - high-grade: from ore containing interlocked pyrite, laboratory investigation, 413
  - production from sulfide ore, 751
- Copper-lead concentrate: separation by sodium dichromate, 754
- Copper milling: Nkana mine, 925
  - Roan Antelope Copper mines, 935
- Copper ores: Anaconda, copper minerals present, 413
- Corundum: mechanical preparation, 788
- Cox, A. B. and Wark, I. W.: *Principles of Flotation, I—Experimental Study of Effect of Xanthates on Contact Angles at Mineral Surfaces*, 189; *Discussion*, 239
  - *Principles of Flotation, II—Experimental Study of Influence of Cyanide, Alkalis and Copper Sulfate on Effect of Potassium Ethyl Xanthate at Mineral Surfaces*, 245
  - *Principles of Flotation, III—Experimental Study of Influence of Cyanide, Alkalis and Copper Sulfate on Effect of Sulfur-bearing Collectors at Mineral Surfaces*, 267
- Crawford, W. E.: *Cyanide Milling Practice of the Fresnillo Company*, 734
- Cromwell, R. H.: *Discussion on Slime-coatings in Flotation*, 411
- Crowe-Merrill precipitation, flow sheet, Witwatersrand, 779
- Crushing (*See also* Ball Milling, Crushing and Grinding, Grinding, Hadsell Mill, etc.): ball milling: gold ore: Pioneer, 679
  - characteristics determined from screen analyses, 146
  - comminution: new methods needed, 124
  - cone: copper ore: Nkana, 928
      - Roan Antelope, 939
    - gold ore: Beattie, 695
      - Kirkland Lake, 652
      - McIntyre Porcupine, 631
      - Pachuca, 722
      - Presidio mine, 711
    - lead-zinc ore: Buchans, 846
  - disk: gold ore: Fresnillo, 735
    - lead-zinc ore: Buchans, 846
  - distribution of particle size, establishment needed, 116
  - efficiency: effect of elasticity of minerals, 93
    - energy equation, 92
    - practical vs. absolute power efficiency, 159
  - elutriation: standardization needed, 119
  - energy equation, 92
  - energy used, summary of investigation, 116
  - gold ore: screen analysis, 153
  - gyratory: copper ore: Nkana, 926
      - Roan Antelope, 939
    - gold ore: Coniauram mill, 619
      - Hollinger mill, 625
      - Kirkland Lake, 651
      - Pachuca, 722
      - Presidio mine, 710
    - lead-zinc ore: Buchans, 846
  - in solution, Black Hills, 607
  - iron ore: Lake Superior, 825
  - jaw: gold ore: Beattie, 695
      - Dome Mines, 621
      - Hollinger mill, 625
      - McIntyre Porcupine, 630
      - Pachuca, 722
    - Tri-State district, 867, 875
  - laws, 90
  - Rittinger theory: confirmed, 116, 146
    - significance, 123
  - rolls: copper ore: Nkana, 926
    - gold ore: Hollinger, 625
    - Polish Bleischarley ores, 954
    - Tri-State district, 867, 875
    - tungsten ore, 834, 836
  - screen analyses: distribution line, 148
  - sedimentation: standardization needed, 119
  - size determination on minus 400-mesh material, simple method needed, 116
  - size distribution of homogeneous material, law, 146
  - stamps: Black Hills, 599, 613
    - Witwatersrand, 762, 771
  - surface on product: direct method for determining needed, 116
    - various methods of measurement, 117
    - various minerals, 120
  - tube milling: Witwatersrand, 764, 771
  - wet: Black Hills, 608
    - history in U.S.: 607
  - work in: bibliography, 124
    - determination, requisites, 117
    - summary of investigation, 116
    - work input and surface produced, 117
- Crushing and grinding: ball milling: gold ore, Siscoe Gold Mines, 567
  - rod milling: Black Hills, 614
    - gold ore with quicksilver, Black Hills, 613
    - phosphate rock, 472 et seq.
- Cryolite: "boiling-over" concentration, 796
- Cuyuna Range: iron-ore beneficiation, 827
- Cyanidation: aeration of solutions, 545
  - after flotation, 700
  - Black Hills, 606

Cyanidation: closely built group of plants affords opportunity for study, 654
  cold climate affects structure of plants at Kirkland Lake, 653
  costs: small mill, Pachuca district, 733
  dissolution of gold: effect of calcium compounds, 675
    rate, effect of alkalinity, 673
  dissolution of gold-silver alloys, rates, 672
  dissolution of gold and silver, 660
    Bodlaender's equations, 666
    measuring electromotive force in gold-cyanide reactions, 664
    measuring loss of gold or silver without removing them from solution, 667, 671
    rate, effect of cyanide concentration, 667, 671
    reactions in dilute solutions, 660
  electromotive force in gold-cyanide reactions, measuring, 664
  Fresnillo, Mexico, 734, 739
  gold ore: before or after amalgamation, respective merits, 538
  high residue avoided by milling in water, 542
  Homestake mill, 607, 614
  impoverishing gold ore before cyanidation justified, 539
  Kirkland Lake district, 646, 649
  McIntyre Porcupine mines, 638
  mercury as an aid to extraction of gold, 538
  oxygen in solutions unaffected by concentration of cyanide, 671
  Pioneer Gold Mines of B. C. Ltd., 678
  Porcupine district, northern Ontario, 617
  precipitation by Crowe-Merrill process, Witwatersrand, 779
  Presidio mill, 704
  reground pyrite concentrate at Noranda, 576
  silver ore, Presidio mill, 704
  with flotation, Kirkland Lake district, 649
  Witwatersrand development, 760 et seq.
Cyanide regeneration, 725, 731, 741
Cyclones: Jumbo, 790
  Laurie, 790
  Pharo, 791
  Torrey, 791

D

Davidson, L. P. and Messner, M. C.: *Concentration of Polish Bleischarley Ores*, 952
Davis, C. W. and Dean, R. S.: *Magnetic Concentration of Ores*, 509
Davis, E. W.: *Iron-ore Beneficiation in Lake Superior District*, 824
Deadwood mill, design, 600
Dean, R. S. and Davis, C. W.: *Magnetic Concentration of Ores*, 509
Debourbage of graphite, 807
del Guidice, G. R. M.: *Microscopy in Flotation Research*, 424
  Study of Slime-coatings in Flotation, 398
del Giudice, G. R. M., Ziehl, O. and Taggart, A. F.: *The Case for the Chemical Theory of Flotation*, 348
DeVaney, F. D., O'Meara, R. G. and Coghill, W. H.: *Advantage of Ball (Rod) Mills of Larger Diameters and Advantages of Improving Bearings*, 79
Dewey, F., Duncan, W. E., Johnson, R. A., Tangel, O. F. Jr. and Gaudin, A. M.: *Reactions of Xanthates with Sulfide Minerals*, 319
Diamonds: mechanical preparation, 805
Diatomite: kinds, 797
  mechanical preparation, 798
  mining, 798
Dolomite: grindability, 62
Dome Mines, Ltd.: gold ore concentration, blanket practice, 621, 623
Dorr, J. V. N. and Anable, A.: *Fine Grinding and Classification*, 161
Drying concentrates: Buchans mill, 852
  Polish Bleischarley ores, 961
Duncan, W. E., Johnson, R. A., Tangel, O. F. Jr., Gaudin, A. M. and Dewey, F.: *Reactions of Xanthates with Sulfide Minerals*, 319
Durham, L. P.: *Nkana Mine Concentrator*, 925
Dust collection: crushing plant, 633

E

Eagle Picher mill: flow sheet, 874
Electrolytic Zinc Co. of Australasia, Ltd., investigation of principles of flotation, 189, 245, 267
Elutriation: pyrite with Nobel elutriator, 589
  summary of research work, 119
Emery: mechanical preparation, 788
Exolon Co.: Johnson separator for kyanite, 809

F

Fahrenwald, A. W.: *Some Fine-Grinding Fundamentals*, 88
Father de Smet mill, 598
Feldspar: grinding costs, 801
  magnetic purification, 800
  mechanical preparation, 799
  milling costs, 801
Filtering: Beattie Gold Mines, 700
  Black Hills, 610
  Buchans mill, 852
  Coniaurum mill, 620
  Fresnillo, 755
  Hollinger Gold Mines, 628
  Kirkland Lake, 652
  Nkana concentrator, 933
  Pachuca, 724
  Pioneer Gold Mines, 685
  Roan Antelope, 948
  Witwatersrand, 768
First Ward mill: cyanidation, 606
Flanagan, V. E.: *Discussions: on Ball Milling*, 74
  on Slime-coatings in Flotation, 411
Florida: phosphate rock occurrence, 815
Flotation: activators needed with xanthates, 211
  adlinear, 350
  absorbed films on solid surfaces, orientation, 237, 242, 243

Flotation: adsorption theories: xanthates with sulfide minerals, 320
  adsorption theory inadequate, 352
  air bubble, constitution, 238
  air-mineral contact: alkali effect on sulfur-bearing collectors, 267
    chalcopyrite: concentrations of depressants to prevent, 294
    copper sulfate effect on sulfur-bearing collectors, 267
    critical pH value, significance, 298
    cyanide effect, 376
    cyanide effect on sulfur-bearing collectors, 267
    in potassium di-n-amyl dithiocarbamate, effect of cyanide, alkalis and copper sulfate, 267
    in potassium ethyl xanthate, effect of cyanide, alkalis and copper sulfate, 267
    in potassium iso-amyl xanthate, effect of cyanide, alkalis and copper sulfate, 267
    in sodium Aerofloat, effect of cyanide, alkalis and copper sulfate, 267
    in sodium di-ethyl dithio-carbamate, effect of cyanide, alkalis and copper sulfate, 267
    in sulfur-bearing collectors, influence of cyanide, alkalis and copper sulfate, 267
    in xanthate solution, effect of cyanide, alkalis and copper sulfate, 267
    influence of cyanide, alkalis and copper sulfate on effect of potassium ethyl xanthate, 245
    pyrite: concentrations of depressants to prevent, 295
    sphalerite: effective depressants, 296
    sulfide minerals in xanthate solution, copper sulfate effect, 245
  albite, 486
  alkalis: effect on air-mineral contact, 245
  almandite, 486
  anorthite, 486
  augite, 486
  barite: soap as reagent, 449
  bauxite: soap as reagent, 449
  biotite, 486
  "boiling-over" concentration of cryolite, 796
  bornite: influence of alkalis and cyanide in xanthate solution, 253, 258
  bubble machine, 192
  bubble test, 491
  cascade cells in ball-mill circuit, 948
  cerussite: removal of xanthate from aqueous solution, 322
  chalcocite: chain formulas of reaction with xanthates, 333, 334, 337, 339
    influence of alkalis and cyanide in xanthate solution, 252
    reaction with xanthates, 328
  chalcopyrite: influence of alkalis and cyanide in xanthate solution, 245

Flotation: chemical theory: argument for and against, 348
  chemical vs. electrostatic theory, 412
  christophite: influence of alkalis and cyanide in xanthate solution, 253
  chromite: soap as reagent, 449
  coatings on sulfide minerals, 323
  contact angle at mineral surfaces: action of alkalis, 217
    air and sulfide minerals in potassium ethyl xanthate, effect of alkalinity and sodium cyanide, 245
    bubble machine, detection of small angles, 234, 241
    characteristic angles, 240
    cyanide effect on sulfide minerals, 221, 237
    drop method of measuring, 493
    effect of method of grinding, 234, 240
    effect of nature of gas used, 223, 236, 243
    effect of various modifying agents, 224
    effect of temperature, 227
    effect of xanthates, 189
    hydrocarbon chain in collector molecules, effect, 189, 233, 240
    measuring, apparatus for, 192
    measuring technique now satisfactory, 233
    preparation of specimen for investigation, 505
    rate of development, 210, 239, 244
    same for all minerals and collectors if nonpolar radical in collector is same, 189, 233
    shape of particles, effect, 210, 234
    significance, 190
  copper: high-grade concentrate from Butte ores: flotation agents consumed, 422
      laboratory investigation, 413
    Nkana mine, 929
    Roan Antelope mill, 946
  copper sulfides: depressant to drop lead, 379
  covellite: influence of alkalis and cyanide in xanthate solution, 253
    reaction with xanthates, 340
  cyanide inhibitory effect destroyed by copper sulfate, 657
  cyanide influence, bibliography, 247
  cyanite, floatability, 486
    soap as reagent, 449
  differential separation of pyrite from pyrrhotite, 571
  electrostatic vs. chemical theory, 412
  epidote, 486
  ferberite: soap as reagent, 449
  floatability of mineral: importance of contact angle, 505
    influence of collecting agent, 499
    relation to density, 488, 505
    requirements, 235, 238
  flocculation: importance, 450, 465
    part in chemical theory, 367
    prevalence, 367
    significance, 367
    to overcome fineness of grinding, 303

- Flotation: fluorspar, 804
  - fluorspar: soap as reagent, 449
  - froth, mineralized, constitution, 239
  - galena: alkali xanthates reaction, 382
    - alkalinity developed with alkali xanthates due to lead carbonate, 388
    - influence of alkalis and cyanide in xanthate solution, 245
    - reaction with xanthates, 324
    - slime-coatings: causes, magnitude and prevention, 398
  - gold: free, recovery, 568
    - pure gold never floated, 374
    - pyrite as carrier, 571, 582
    - requisite conditions, 374
    - with cyanidation, 649
  - graphite, 807
  - hypothesis for nonflotation of sulfide minerals of near-colloidal size, 303
  - inherent floatability, 369
  - iron oxide, experiments, 830
  - kyanite, 810
  - labradorite, 486
  - lead-copper concentrate separated by sodium dichromate, 754
  - lead-zinc ore, Buchans, 849
  - limestone: soap as reagent, 449
  - manganese oxides: soap as reagent, 449
  - marmatite: influence of alkalis and cyanide in xanthate solution, 253
  - mineral surfaces: old and new, differences in reactions, 304
    - preparation for investigation in laboratory, 194
  - muscovite, 486
  - nephelite, 486
  - nonmetallic minerals: chemical theory, 375
  - Noranda, 571, 582
  - olivine, 486
  - orthoclase, 486
  - Ostwald theory refuted, 350
  - overgrinding of ore cause of loss of metals, 303
  - pH control, 380
  - phosphate rock: Phosphate Recovery Corporation, Florida: development of concentration, 480
      - No. 2 concentrator, 466
    - soap as reagent, 449
  - preceded by thickening: complex ore producing lead, copper, zinc and iron concentrates, 751
  - principles: bibliography, 231
    - investigation at University of Melbourne, 189, 245, 267
  - pyrite: influence of alkalis and cyanide in xanthate solution, 245
    - reaction with xanthates, 341
  - pyrite and pyrrhotite: increasing gold recovery at Noranda, 578
  - pyrrhotite for recovery of gold, 577
  - quartz, 486
  - regrinding and refloating: copper ore, 931
    - hard gold ore, 690
  - research: microscope as tool for investigation, 424
- Flotation: rhodochrosite: soap as reagent, 449
  - rhodonite, 486
  - scheelite: soap as reagent, 449
  - siderite: soap as reagent, 449
  - silicate minerals: floatability: causes, 486
      - formula for calculating, 503
      - importance, 486
      - relative, 486
  - slime-coatings: bibliography, 410
    - chemical hypothesis, 399
    - grinding of ore a cause, 410
    - magnitude, 408
    - mechanism, theory, 398
    - microscopy in examination, 400, 424
    - prevention, 380, 406
    - responsible for nonflotation, 305
  - soap as reagent with nonsulfides, 449
  - soap: role, 451
  - sodium cyanide: effect on air-mineral contact, 245
  - sphalerite: influence of alkalis and cyanide in xanthate solution, 245
  - spodumene, 486
  - sulfide minerals: coatings on, 323
    - near-colloidal size: effect of adding reagent before and after grinding, 308
      - inert coatings cause of poor flotation, 303
      - nonflotation, hypothesis for, 303
    - reaction with xanthates, 319
    - xanthating, chemical-reaction theory, 323
    - with xanthate: adsorption theories, 320
  - sulphydryl radical: orientation, 393
  - titanite, 486
  - topaz, 486
  - tourmaline, 486
  - tremolite, 486
  - wettability of mineral: definition, 488
    - relation to density and melting point, 495, 497
  - with cyanidation, 700
  - with magnetic separation and magnetizing roasting of complex tungsten ore, 833
  - xanthates: activators needed, 211
    - effect on contact angles at mineral surfaces, 189
    - reaction with sulfide minerals, 319
    - removal from aqueous solution by cerussite, 322
  - xanthates, alkali: action on galena, 382
    - alkalinity developed with galena due to lead carbonate, 388
  - zinc-lead ores, 957, 960
    - Tri-State district, 890
  - zircon, 486
- Flotation reagents: activators for use with xanthate, 211
  - alkali xanthates: stability, 395
  - collectors: for use with xanthate, 215, 216
  - soap, for nonsulfides, 449
  - soap: Lifebuoy, 651
- Flow sheets: copper milling: Roan Antelope Copper Mines, 938

Flow sheets: copper milling: Symons plant, Roan Antelope mine, 942
    Nkana concentrator, 927
  Crowe-Merrill precipitation, Witwatersrand, 779
  crushing: Hollinger Gold Mines, 626
    McIntyre Porcupine Mines, 631
  cyanidation: Beattie Gold Mines, 702
    Fresnillo, 746
    Hollinger Gold Mines, 627
    McIntyre Porcupine Mines, 638
  flotation: Fresnillo concentrator, 753
    McIntyre Porcupine Mines, 634
  gold milling: Beattie Gold Mines, 698
    Coniaurum Mines, 618
    Dome Mines, 622
    McIntyre Porcupine, 633
    Montezuma-Apex Mining Co., 564
    Noranda concentrator, 581
    Pioneer Gold Mines, 682
    Siscoe Gold Mines Ltd., 567
    Teck-Hughes mill, 656
    Witwatersrand, 763 et seq.
    Wright-Hargreaves mill, 658
  grinding, conversion from two-stage to single-stage, Roan Antelope, 944
  lead-zinc milling, Buchans mill, 844
  Phosphate Recovery Corporation, 471 et seq.
  sulfur dioxide treatment: Fresnillo, 748
  tabling and cyanidation, Presidio Mine, 708
  tungsten concentrator, 837
  zinc-lead ore: Ballard mill, Tri-State district, 868
    Bird Dog mill, 872
    Eagle Picher mill, 874
    flotation, 958
    gravity concentration, 955
    Scott mine, Tri-State district, 870
Fluorspar: flotation, 804
  mechanical preparation, 801
Fresnillo Company: flotation preceded by thickening: complex ore of lead, copper zinc, gold and silver, 751
  milling practice, gold-silver ores, 734
  tailings reclamation, 742
Fresnillo district, Mexico: geology, 734
Fuller's earth: mechanical preparation, 804

G

GAHL, R.: *Discussion on Principles of Flotation*, 233
Galmei, 952
Garnet abrasive: mechanical preparation, 788
GAUDIN, A. M.: *Discussions: on Principles of Flotation*, 233
  on Soap Flotation of the Nonsulfides, 464
GAUDIN, A. M., DEWEY, F., DUNCAN, W. E., JOHNSON, R. A. AND TANGEL, O. F. JR.: *Reactions of Xanthates with Sulfide Minerals*, 319
GAUDIN, A. M. AND MALOZEMOFF, P.: *Hypothesis for Nonflotation of Sulfide Minerals of Near-colloidal Size*, 303
GAYFORD, E.: *Ore Treatment as a Factor in Small Gold-mining Enterprises*, 549
Gemstones: mechanical preparation, 805
Geophysical prospecting: discovery of lead-zinc orebodies, Buchans mine, 841
GEORGE, P. W. AND HELLSTRAND, G. A.: *Milling Practice at Buchans Mine, Buchans, Newfoundland*, 841
Giesche Spólka Akcyjna: concentration of zinc-lead ores, 952
Glasspar: graded crushing, 800
Gold: Black Hills: history, 597
  Black Hills: metallurgy, review, 597
  bullion for U. S., terms and conditions, 549
  colloidal: color changes, 544
  value on basis of monetary dollar, 547, 549
Gold metallurgy: technical accountancy, 547
Gold milling (*See also* Cyanidation, Flotation, etc.): Beattie Mines, 690
  Black Hills, 597
  blanket practice, Dome Mines, 621, 623
  blanket strakes for recovery of free gold, 561, 567, 577, 601, 775
  blanket table, self-washing, 562
  bulk leaching: economical techniques, 539
  choice of method, 553
  classification of sand tailing, Homestake mills, 609
  double treatment of sand and slime, benefits, 542
  drying before hydrometallurgical treatment advisable, 543
  economical techniques, 538
  extraction vs. recovery, 539
  factor in small gold mining enterprises, 549
  flotation followed by cyanidation, 630
  free gold recovery, 561, 568, 577, 601, 621 623, 775
  Fresnillo Company, 734
  gravity concentration of sulfides for intensive cyanidation, 624
  grinding in cyanide solution, 624, 680
  hard ore, 690
  Homestake, 597
  hydrometallurgical treatment: colloidal gold cause and prevention, 544
    time factor important, 541
  in cyanide solutions or in water, 541
  in water, advantages ,542
  Kirkland Lake district, 649
  Montezuma-Apex Mining Co., 563
  Noranda, 570
  Pachuca, 732
  Pioneer Gold Mines of B.C., Ltd., 678
  Porcupine district, northern Ontario, 617
  Presidio mill, 710
  pyrite ore, 690
  pyrite and pyrrhotite: copper recovery, 578
    increasing gold recovery at Noranda, 570
    Witwatersrand, 760
  pyrite regrinding, Noranda, 582
  regrinding flotation concentrate, 575, 582
  sampling of ore important, 554
  sand tailing treatment, Black Hills, 609
  Siscoe Gold Mines, Ltd., 566
  sizing at Homestake, 611
  slime decantation process, Witwatersrand, 768

Gold milling: small mills: choice of method, 553
    sulfide ore, Witwatersrand, 760
    tailings reclamation, 742
    tellurides mean fine grinding, 646
    Witwatersrand, development, 760
Gold mining: equipment, 560
    expert advice necessary, 549
    power supply, 558
    producing bullion at mine vs. shipping a concentrate, 550
    small enterprises: ore treatment as factor, 549
    transportation, 559
    water supply important, 558
Gold ore: banket, 760
    microscopic investigation at Noranda, 572
    milling. *See* Gold Milling.
    Montana, screen analysis, 153
    Noranda: mineralogical analysis, 570
    pyrite: sizing with Nobel elutriator, 573, 589
    quantitative determination of pyrite and pyrrhotite in ore and mill products at Noranda, 593
    treatment, economical techniques, 538
    treatment as factor in small mining enterprises, 549
    unit in reporting assays, 547
    Witwatersrand: size of particles, 761
Gold refining: Pioneer Gold Mines, 685
Gold smelting: average schedule and application to concentrates, 556
    Black Hills, 605
Gold trap, hydraulic, 561, 568
Golden Gate plant: cyanidation, 606
Golden Reward Co., amalgamation in Black Hills, 604
Golden Star mill, design, 600
Golden Terra mill, design, 600
Gow, A. M., Guggenheim, M., Campbell, A. B. and Coghill, W. H.: *Ball Milling*, 24
Granby Consolidated Mining, Smelting and Power Co.: grinding flow sheet, 171
Granite: mechanical preparation, 820
Granules for roofing, 821
Graphite: mechanical preparation, 806
Gravel: mechanical preparation, 818
Gravity concentration: gold ore, Montezuma-Apex, 564
        Pachuca, 723
        Siscoe Gold Mines, 566
    Hollinger Gold Mines, 628
        jigging: iron ore, 826
        zinc-lead ores, 876, 956
    Polish Bleischarley ores, 954, 956
    Presidio gold mine, 712, 715
    silver-gold-lead ore, for recovery of lead, 715
    Tri-State district, 867, 876, 905
    tungsten ore, 836
Gray, F. E. and Howbert, V. D.: *Milling Methods and Costs at Presidio Mine of American Metal Co. of Texas*, 704
Grinding (*See also* Ball Milling, Crushing, Hadsel Mill, etc.): amount of horsepower in U.S., 24
    ball milling: copper ore: Nkana, 928
            Roan Antelope: 943
        gold ore: Pioneer, 680
            Beattie, 697
            Fresnillo, 736
            Kirkland Lake, 652
            Montezuma-Apex, 564
            Pachuca, 722
        lead-copper ore: Fresnillo, 751
        lead-zinc ore: Buchans, 847
            Polish, 959
        zinc-lead ores: Tri-State district, 889, 910
        Witwatersrand, 766
    characteristics determined from screen analyses, 146
    closed-circuit, 106
        circulating load, advantage of high, 173
        classifier importance, 171
        examples, 171
        grindability of ore: determining, 131
        grindabilities of various ores, 130
        increase of mill capacity, 171
        progressive changes, 175
    copper ore: typical flow sheets, 170
    evaluating screen analysis by calculating diameter of median gram, 136
    feed: load, effect on efficiency, 103
        size, effect on output, 101
    fine: classifier as important as mill, 161
        closed-circuit, 166
            evolution of system, 168
            typical flow sheets, 169
        critical speed, 94
        efficient plant, requirements, 176
        fundamentals, 88
        high-speed, mill design and operating conditions, 95
        interdependence of rotary mill and classifier, 161
        mill efficiency, 166
        open-circuit, 165
        plant, requirements for efficiency, 176
        points of commercial interest, 89
        point where crushing terminates, 88, 101
        power consumption, 171
        relation to classifier efficiency, 106
        relation of fineness of grinding to mill capacity, 113
    fineness of product in relation to grinding rate, 113
    gold ore: reagents in circuit beneficial, 699
    grindability of ore: determining, 131
        heterogeneous ores, 57
        various ores, 91, 130
        wet and dry, 140
    hard-grinding fraction found by screen analyses, 146
    loss of metals caused by overgrinding characteristic of very fine particles, 303
    multiple-stage, 168
    presence and location of natural grain sizes found by screen analyses, 146
    primary and secondary circuits to produce high-grade flotation concentrate from copper ore, 413
    pulp density, effect on mill performance, 104

- Grinding: rate of grinding: relation to fineness of grinding, 113
  - regrinding: copper concentrates, 931
    - pyrite to increase gold recovery, 575, 582
  - rod milling: gold ore: Fresnillo, 736
      - Hollinger, 627
      - Pachuca, 722
    - lead-copper ore: Fresnillo, 751
  - suitability of media found by screen analyses, 146
  - time of residence of pulp in mill, formula for determining, 105
  - tube milling: gold ore: Beattie, 697
      - Coniaurum mill, 619
      - Dome Mines, 621
      - Hollinger, 627
      - Kirkland Lake, 651
      - McIntyre Porcupine, 633
      - Presidio, 712
    - pieces of ore used as pebbles, Witwatersrand, 764
  - work done: circulating load, 167
  - work in: Kick theory, 166
    - Rittinger theory, 166
- Grinding mill, definition, 88
- Grinding mills: commercial: classification, 90
  - comparison, 164
  - grinding media, important factors, 99
  - high-speed: design and operating conditions, 95
  - mechanical elements, 88
  - variables that affect performance, 90
- Griswold, G. G. Jr. and Morrow, B. S.: *Production of High-grade Concentrate from Butte Copper Ores—Results of Laboratory Investigations*, 413
- Gross, J.: *Summary of Investigation on Work in Crushing*, 116
- Guess, H. A.: lead-zinc milling at Buchans mine, 841
- Guggenheim, M., Campbell, A. B., Coghill, W. H. and Gow, A. M.: *Ball Milling*, 24
- Gypsum: mechanical preparation, 808

H

- Hadsel mill: construction and operation, 15
  - stage grinding, 62
- Halite: separation from complex salts, 817
- Hall, R. G.: *The Hadsel Mill*, 15
- Hamilton, P. D. P.: *Milling Methods of Porcupine District of Northern Ontario*, 617
- Hancock, R. T.: *Discussion on Ball Milling*, 76
- Harbaugh, M. D., Burris, S. J. Jr., Anderson, C. O., Illidge, R. E. and Howes, W.: *Milling Practice in Tri-State Zinc-lead Mining District*, 861
- Harris Clay Co.: mica preparation, 813
- Hedley, N., Barsky, G. and Swainson, S. J.: *Dissolution of Gold and Silver in Cyanide Solutions*, 660
- Heizer, O. F.: *Concentration of Tungsten Ore by the Nevada-Massachusetts Co.*, 833
- Hellstrand, G. A. and George, P. W.: *Milling Practice at Buchans Mine, Buchans, Newfoundland*, 841
- Hematite: powdered: magnetic constants, 519, 523
- Hey, H.: cooperation in investigation of principles of flotation, 189, 245, 267
- Highland mill, design, 600
- Hollinger Consolidated Gold Mines, Ltd.: gold ore concentration, 624
  - milling costs, 625, 629
- Homestake (*See also* Black Hills): district: metallurgy review, 597
  - mills: blankets, 601
    - bullion as shipped, 615
    - cyanidation, 607
    - milling, review, 597
    - sand tailing classification, 609
  - mine: history, 597
- Horse patio, Loreto, Mexico, 726
- Howbert, V. D. and Gray, F. E.: *Milling Methods and Costs at Presidio Mine of American Metal Co. of Texas*, 704
- Howes, W., Harbaugh, M. D., Burris, S. J. Jr., Anderson, C. O. and Illidge, R. E.: *Milling Practice in Tri-State Zinc-lead Mining District*, 861
- Hubler, W. G. and Martin, F. J.: *Ore Treatment at Beattie Gold Mines Ltd.*, 690
- Hutchinson, W. S.: Appreciation of Robert H. Richards, 11
- Hydraulic gold trap, 561, 568

I

- Illidge, R. E., Howes, W., Harbaugh, M. D., Burris, S. J. Jr. and Anderson, C. O.: *Milling Practice in Tri-State Zinc-lead Mining District*, 861
- Ilmenite: powdered: magnetic constants, 519, 523
- Iron: sponge. *See* Sponge Iron.
- Iron concentrate: produced by flotation from complex ore for its gold and silver values, 751
  - smelting, 831
  - sponge iron production, experiments, 831
- Iron-ore beneficiation: crushing and screening, 825
  - definition, 824
  - drying and sintering, 827
  - Hydrotator separation, 828
  - jigging, 826, 829
  - Lake Superior district, 824
  - low-grade ore in Lake Superior district, 824
  - magnetic concentration, 826
  - processes, classification, 824
  - Rheolaveur separation, 828
  - washing, 825
- Iron-ore roasting: magnetic, 830
- Irvin, D. F.: *Cyanidation at Kirkland Lake*, 646

J

- Jigging. *See* Gravity Concentration.
- Jigs: Bendelari diaphragm, 878
  - Cooley, 878
  - Smith and Maxwell, 775
- Job cone classifier, 772
- Johnson concentrator, 775

Johnson induction magnetic separator for kyanite, 809
Johnson, O. H.: *Discussion on Advantage of Ball (Rod) Mills of Larger Diameters and Advantage of Improving Bearings*, 86
Johnson, R. A., Tangel, O. F. Jr., Gaudin, A. M., Dewey, F. and Duncan, W. E.: *Reactions of Xanthates with Sulfide Minerals*, 319
Jumbo cyclone, 790

K

Kaolin: mechanical preparation, 795
Kick law. *See* Crushing and Grinding.
Kildonan mill, 605
Kirkland Lake district: engineers in charge of mills, 659
  gold-ore treatment: Kirkland Lake Gold mill, 649
   Lake Shore mill, 649
   Macassa mill, 649
   Sylvanite mill, 649
   Teck-Hughes mill, 649, 655
   Toburn mill, 649
   Wright-Hargreaves mill, 649, 657
  ore: grade, 647
  production statistics, 649
Kirkland Lake Gold mill. *See* Kirkland Lake district.
Knoll, A. F. and Taylor, T. C.: *Action of Alkali Xanthates on Galena*, 382
Kuryla, M. H. and Bryan, R. R.: *Milling and Cyanidation at Pachuca*, 722
Kyanite: mechanical preparation, 808
  mica concentrates joint product from schists, 810

L

Lake Shore mill. *See* Kirkland Lake.
Lake Superior district: iron-ore beneficiation, 824
  future of iron-ore industry, 832
  probable effect of St. Lawrence Waterway, 828
Laurie cyclone, 790
Leaching: bulk: gold ores, economical techniques, 539
  Cuyuna ores for manganese, 830
  Homestake ore, 541
Lead: flotation concentrate: production from sulfide ore, 751
  recovery from silver-gold ore, Presidio mill, 710
Lead-copper concentrate: separation by sodium dichromate, 754
Lead-zinc ore: milling at Buchans, 841
Legrand, C.: *Discussion on Ball Milling*, 75
Legsdin, A.: *Discussions: on Relative Floatability of Silicate Minerals*, 507
   on Slime-coatings in Flotation, 412
Lepidocrocite: hysteresis data, 524
  powdered: magnetic constants, 519, 523
Limestone: mechanical preparation, 819
Littleford, J. W.: *Concentration Operations at Roan Antelope Copper Mines, Ltd.*, 935
Locke, C. E.: *Preface*, 5
  Discussions: on Ball Milling, 73
   on Relative Floatability of Silicate Minerals, 507
Lodestone: powdered: magnetic constants, 519
Loreto, Mexico, milling and cyanidation, 722
Lucky Strike: ore, 848
  orebody, discovery, 841
Lundberg, H.: geophysical discovery of orebodies at Buchans mine, 841

M

Macassa mill. *See* Kirkland Lake.
MacLean classifier, 772
Madagascar: graphite preparation, 807
Magna mill. *See* Utah Copper Co.
Magnesite: mechanical preparation, 810
Magnetic concentration: development of art, 509
  iron-ore beneficiation, Lake Superior district, 826
  magnetic properties of minerals, determining, 513
  magnetite from Richard mine, Wharton, N. J., 527
  pretreatment of minerals, 520
  separation with flotation and magnetizing roasting of complex tungsten ore, 833
  separators. *See* Magnetic Separators.
Magnetic properties of minerals, determining, 513
Magnetic roasting of iron ore, 830
Magnetic separators: classification, 510
  commercial: field and limitations at present, 526
  new type based on coercive force and remanence, 530
  new type based on repulsion, 533
  patents issued, 510
Magnetite: magnetization curve, 518
  powdered: magnetic constants, 519, 523
  Richard mine, Wharton, N. J.: magnetic concentration, 527
  Ural Mountain: hysteresis data, 521
Malozemoff, P. and Wilkinson, W. D.: *Discussion on Soap Flotation of the Nonsulfides*, 465
Malozemoff, P. and Gaudin, A. M.: *Hypothesis for Nonflotation of Sulfide Minerals of Near-colloidal Size*, 303
Manganese: recovery from Cuyuna ores by leaching, 830
Manganese-silver ore: treatment with sulfur dioxide, 744
Marble: mechanical preparation, 819
Martin, H. S.: *Milling Methods and Costs at No. 2 Concentrator of Phosphate Recovery Corporation, with Appendix on New Developments in Flotation Concentration of Phosphate Rock*, 466
Martin, F. J. and Hubler, W. G.: *Ore Treatment at Beattie Gold Mines Ltd.*, 690
Maxson, W. L.: *Discussion on Advantage of Ball (Rod) Mills of Larger Diameters and Advantage of Improving Bearings*, 85

Maxson, W. L. and Bond, F. C.: *Crushing and Grinding Characteristics as Determined from Screen Analyses*, 146
Maxson, W. L., Cadena, F. and Bond, F. C.: *Grindability of Various Ores*, 130
Maxwell. *See* Smith.
McIntyre Porcupine Mines, Ltd.: analyses of ore and products, 635 et seq.
  cyanidation costs, 642
  gold ore concentration, 630
  milling costs, 630, 633
McLachlan, C. G.: *Increasing Gold Recovery from Noranda's Milling Ore*, 570
McLanahan-Watkins Co.: kyanite preparation, 809
Mechanical preparation of nonmetallic minerals, 785
Meerschaum: mechanical preparation, 810
  occurrence, New Mexico and Turkey, 810
Mesabi Range: iron-ore beneficiation, 825
Messner, M. C. and Davidson, L. P.: *Concentration of Polish Bleischarley Ores*, 952
Metallurgical complications: prevention better than cure, 543
Mexico. *See* Fresnillo; Pachuca
Miami Copper Co.: grinding flow sheet, 170
Mica: mechanical preparation, 811
Mica concentrates: joint product with kyanite from schists, 810
Microscope: Abbe substage, 428
  compound: optical systems, 425
  eyepieces, 442
  illumination: Cardioid condenser, 445
    incident, transmitted and vertical, 443
    Ultropak, 400, 424, 444
  in flotation research, 424
  magnification and numerical aperture, 428
  objectives, 436
  oculars, 442
  Ramsden circle, 442
  specimens: finger-smeared, defects, 447
  specimens: preparation, 400, 445
  tests of gold ore at Noranda, 572
Mills (see also Ball Mills, Rod Mills, Tube Mills): Hadsel, construction and operation, 15
Mineral powders. *See* Powdered Minerals.
Montezuma-Apex Mining Co.: milling gold ore, 563
Monazite: mechanical preparation, 815
Morrow, B. S. and Griswold, G. G. Jr.: *Production of High-grade Concentrate from Butte Copper Ores—Results of Laboratory Investigations*, 413
Morton, R. J.: work on increasing gold recovery from Noranda's milling ore, 589, 593
Mount Lyell Mining & Railway Co.: investigation of principles of flotation, 189, 245, 267

N

Nevada Consolidated Copper Co.: grinding flow sheet in 1929, 170
Nevada-Massachusetts Co.: concentration of tungsten ore, 833
New Mexico: meerschaum occurrence, 810
Newton, H. W : *Discussion on Ball Milling*, 74
Nkana mine concentrator: crushing, 925
  flotation, 929
    reagents, 932
  grinding, 928
  power consumption, 933
  regrinding, 931
Nobel elutriator: description, 589
  sizing finely ground pyrite, 573, 589
Nonmetallic minerals: economic importance, 786
  mechanical preparation, 785
  milling, 785
Noranda Mines, Ltd.: concentrator flow sheet, 580
  cyanidation of reground concentrate, 576, 588
  flotation concentration of gold ore, 571, 582
  increasing recovery from milling ore, 570
  microscopic investigation of gold ore, 572
  ore, mineralogical analysis, 570
  quantitative determination of pyrite and pyrrhotite in ore and mill products, 593
  regrinding pyrite concentrate for recovery of gold, 575, 582
Northwest Magnesite Co.: preparation of magnesite, 810

O

O'Meara, R. G., Coghill, W. H. and DeVaney, F. D.: *Advantage of Ball (Rod) Mills of Larger Diameters and Advantage of Improving Bearings*, 79
Oriental orebody: discovery, 841
Osmiridium: recovery in milling sulfide ores Witwatersrand, 760

P

Pachuca district, Mexico: costs at small cyani mill, 733
  cyanidation developments, 1907–1934, 722
  gold recovery, 732
  milling methods, developments in mills, 722
  silver recovery, 732
Pachuca tank, 724
Paint pigments. *See* Pigments.
Patek, J. M.: *Relative Floatability of Silicct Minerals*, 486; *Discussion*, 507, 508
Pharo cyclone, 791
Phosphate Recovery Corporation: development of flotation concentration, 480
  flotation concentration of washer waste, 480
  milling methods and costs at No. 2 concentrator, Mulberry, Florida, 466
  operating costs, 479, 483
Phosphate rock: flotation. *See* Flotation.
  mechanical preparation, 815
  mining: hydraulic guns, pebble district, Florida, 466
    suction dredge, pebble district, Florida, 481
  occurrence in Florida and Tennessee, 815
  washing, pebble district, Florida, 467
Pigments: mechanical preparation, 814
Pioneer Gold Mines of B.C. Ltd.: milling methods, 678
  production figures, 678
  refining, 688

Polish Upper Silesia: Bleischarley zinc-lead ores, concentration, 952
Porcupine district, northern Ontario: milling methods: Coniaurum Mines, 617
  Dome Mines, 621
  Hollinger mill, 624
  McIntyre Porcupine Mines, 630
Powdered minerals, various, magnetic constants, 519, 523
Presidio mill: conveying, 719
  cyanidation, 717
  gold recovery, 710, 711
  lead recovery, 710
  methods of milling silver-gold-lead ore, 704
  milling costs, 704
  sampling, 720
  silver recovery, 707, 710, 711
Pulp: ore: colloidal material, amount, 181
    compression, or thickening, 185
    flocculent, behavior, 183, 186
    flocculent: colloidal aspects, 187
    flocculation, methods and mechanism of obtaining, 178
    settling rate, 182
    thickening, colloidal chemistry, 178
Pumicite: mechanical preparation, 788
Pyrite (*See also* Gold Milling and Gold Ore):
  mechanical preparation, 821
  powdered: magnetic properties, effect of heat treatment, 523
Pyritic smelting: Black Hills, 605
Pyrophyllite: mechanical preparation, 817
Pyrrhotite (*See also* Gold Milling and Gold Ore):
  powdered: magnetic constants, 519

R

Ramsden circle. *See* Microscope.
Rhokana Corporation: Nkana mine concentrator, 925
Richards, R. H.: achievements and service in mechanical concentration of ores, 11
Rittinger law. *See* Crushing and Grinding.
Roan Antelope Copper Mines Ltd.: concentrating operations, 935
  flotation, 946
  mill: costs, 947, 950
    crushing and grinding, 939
    tailings disposal, 949
  mill-feed assays, 937
  research work, 950
Roberts, E. J.: *Colloidal Chemistry of Pulp Thickening*, 178
Robinson, B.: work on increasing gold recovery from Noranda's milling ore, 593
Rock Candy mill: fluorspar preparation, 803
Rod mills (*See also* Grinding Mills):
  uses, 165
Rossiter plant: cyanidation, 606
Rottenstone: mechanical preparation, 788

S

St. Lawrence Deep Waterway: probable effect on Lake Superior district iron and steel industry, 828
St. Louis Smelting & Refining Co.: Ballard mill flow sheet, 868
Salines: mechanical preparation, 817
Sampling: Presidio mill, 720
Sand: mechanical preparation, 818
Sandstone: mechanical preparation, 819
Santa Gertrudis, milling and cyanidation, 722
Schutz, P. and Spry, R.: *Milling Gold Ores at Pioneer*, 678
Screen analyses: distribution line, 148
  evaluating, 136
Silicon carbide: mechanical preparation, 788
Silver-gold-lead ore: gravity concentration, Presidio mill, 715
Silver milling: Fresnillo Company, 734
  manganiferous ores: sulfur dioxide process, 744
  methods at Presidio mill, 704
  recovery, Pachuca district, 732
  recovery at Presidio mill, 707, 710, 711
  tailings reclamation, 742
Sintering: iron ore: Lake Superior district, 827
Siscoe Gold Mines Ltd., milling gold ores, 566
Slate: mechanical preparation, 820
Slime-coatings. *See* Flotation.
Smelting. *See* Pyritic.
Smith and Maxwell jig, 775
Sorting: Polish Bleischarley ores, 956
  Witwatersrand, 770
Sponge iron: from concentrates, experiments, 831
  magnetization curve, 518
Spry, R. and Schutz, P.: *Milling Gold Ores at Pioneer*, 678
Steel: powdered: magnetic constants, 519
Stokes law. *See* Classification.
Stone: mechanical preparation, 819
Sulfur: mechanical preparation, 821
Sulfide ore: quantitative determination of pyrite and pyrrhotite in Noranda ore and mill products, 593
Sulfur dioxide treatment for manganiferous silver ores, 744
Swainson, S. J., Hedley, N. and Barsky, G.: *Dissolution of Gold and Silver in Cyanide Solutions*, 660
Sylvanite mill. *See* Kirkland Lake.
Sylvite: separation from complex salts, 817

T

Table treatment. *See* Gravity Concentration.
Taggart, A. F.: *Discussions: on Relative Floatability of Silicate Minerals*, 505, 506
  on Slime-coatings in Flotation, 411, 412
Taggart, A. F., del Giudice, G. R. M. and Ziehl, O.: *The Case for the Chemical Theory of Flotation*, 348
Tailings reclamation: gold ore: Fresnillo, 742
Talc: mechanical preparation, 822
  pyrophyllite, 817
  value per ton, 822
Tangel, O. F. Jr., Gaudin, A. M., Dewey, F., Duncan, W. E. and Johnson, R. A.: *Reactions of Xanthates with Sulfide Minerals*, 319

TAYLOR, T. C. AND KNOLL, A. F.: *Action of Alkali Xanthates on Galena*, 382
Teck-Hughes mill. *See* Kirkland Lake.
Tennessee: phosphate rock occurrence, 815
Tennessee Minerals Products Co.: feldspar, mechanical preparation, 799
Thickening (*See also* Pulp):
  Coniaurum mill, 619
  Fresnillo, 755
  Hollinger Gold Mines, 629
  Kirkland Lake, 652
  Pachuca, 723
  Pioneer Gold Mines, 683
  Polish Bleischarley ores, 961
  Presidio mill, 712
  pulp: colloidal chemistry, 178
  Tri-State district, 885
  Witwatersrand, 764
Thoen Stone, 597
THOMAS, E.: *Discussion on Relative Floatability of Silicate Minerals*, 506
Toburn mill. *See* Kirkland Lake.
Torrey cyclone, 791
Tramway, shuttle, 566
Tripoli: mechanica preparation, 788
Tri-State zinc-lead district: ball milling, 889, 910
  buying and selling of concentrates, 920
  mill-construction costs, 913
  mill crew, 914
  mill operating costs, 914 et seq.
  milling practice: chat-middling circuit, 881
    chat-roughing jigs, 884, 909
    cleaner jigging, 880, 907, 908
    concentrate disposal, 900
    crushing and screening, 875
    flotation, 890
    metallurgical results, 903
    ore storage and conveyance, 869
    power supply and requirements, 902
    rougher jigging, 876, 905, 906
    tables, arguments for and against, 888
    tabling, 885, 909
    tailings disposal, 899, 913
    tailings re-treatment, 917
    trend, 921
    wage scale, 914
    water supply and requirements, 901
  prices for zinc and lead concentrates, 865
  zinc-lead ores milled, 863
Tube mills: uses, 165
Tungsten ore: concentration by flotation, magnetic separation and magnetizing roasting, 833
  concentration by Nevada-Massachusetts Co.: analysis of mill feed, 834
    flow sheet, 835
    production statistics, 835
Turkey: meerschaum occurrence, 810
TYLER, P. M.: *Mechanical Preparation of Nonmetallic Minerals*, 785

U

Ultropak. *See* Microscope.
U. S. Bureau of Mines: investigation on work in crushing, 116
U. S. Bureau of Mines: measurement of surface of quartz, 117
United Verde Copper Co.: grinding flow sheet, 170
University of Melbourne: investigation of principles of flotation, 189, 245, 267
Utah Copper Co.: Magna mill: grinding flow sheet in 1929, 170

V

Vermiculite: mechanical preparation, 823
  Zonolite by Schundler process, 823
Vermont: asbestos yield, 791
Vitrefrax Corporation: kyanite preparation, 809
VON BERNEWITZ, M. W.: *Discussion on Ball Milling*, 75

W

WARK, I. W. AND COX, A. B.: *Principles of Flotation, I—Experimental Study of Effect of Xanthates on Contact Angles at Mineral Surfaces*, 189; *Discussion*, 239
  Principles of Flotation, II—Experimental Study of Influence of Cyanide, Alkalis and Copper Sulfate on Effect of Potassium Ethyl Xanthate at Mineral Surfaces, 245
  Principles of Flotation, III—Experimental Study of Influence of Cyanide, Alkalis and Copper Sulfate on Effect of Sulfur-bearing Collectors at Mineral Surfaces, 267
WARTENWEILER, F.: *Development of Milling and Cyanidation on the Witwatersrand*, 760
Whiting: mechanical preparation, 794
WILKINSON, W. D. AND MALOZEMOFF, P.: *Discussion on Soap Flotation of the Nonsulfides*, 465
Witwatersrand: banket ore, 760
  decantation of slime, 768
  milling and cyanidation: bibliography (brief), 784
  milling and cyanidation development, 760
  milling costs, 782
  milling flow sheets, 763 et seq.
  mines, 781
  operating groups, 781
  recoveries, 780
  tonnages, 780
Wright-Hargreaves mill. *See* Kirkland Lake.

X

Xanthates. *See* Flotation.

Z

ZIEHL, O., TAGGART, A. F. AND DEL GIUDICE, G. R. M.: *The Case for the Chemical Theory of Flotation*, 348
Zinc: flotation concentrate: production from sulfide ore, 751
Zinc-lead ores: Polish Upper Silesia: concentrating, 952
  Tri-State milling practice, 861
Zonolite. *See* Vermiculite.